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9950-632
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() LOW COST CZOCHRALSKI CRYSTAL GROWING
TECHNOLOGY. NEAR IMPLEMENTATION OF THE FLAT
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COST SOLAR ARRAY PROJECT Final Report, 12
Mar. 1979 - 30 Sep. (Kayex Corp., Rochester, G3/44 16814

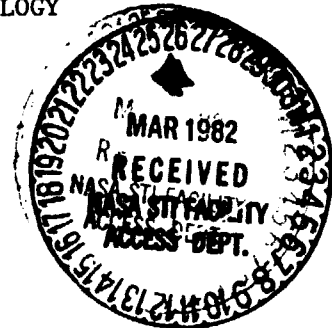
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LOW COST CZOCHRALSKI CRYSTAL GROWING TECHNOLOGY

NEAR TERM IMPLEMENTATION
OF THE
FLAT PLATE PHOTOVOLTAIC COST REDUCTION
OF THE
LOW COST SOLAR ARRAY PROJECT



FINAL REPORT
MARCH 12, 1979 - SEPTEMBER 30, 1980
PROGRAM MANAGER: R. L. LANE
PRINCIPAL INVESTIGATOR: E. G. ROBERTS

KAYEX CORPORATION
1000 MILLSTEAD WAY
ROCHESTER, NEW YORK 14624

"The JPL Low Cost Silicon Solar Array Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of low cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, by agreement between NASA and DOE."

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ACKNOWLEDGEMENTS

The author wishes to acknowledge and express appreciation to the persons who were instrumental in completing the work undertaken in this contract.

Dr. R. L. Lane - Project Manager - Kayex Corp., Rochester, NY

Dr. R. Kachare and A. Morrison - JPL Technical Project Managers

M. Larson - JPL Contract Negotiator Specialist

N. Sarno - Process Engineer - Kayex Corp., Rochester, NY

M. Johnson - Process Engineer - Kayex Corp., Rochester, NY

D. Rakover - Assistant Process Engineer - Kayex Corp., Rochester, NY

D. Ewen and P. Eckardt - Process Technicians - Kayex Corp., Rochester, NY

D. Fanale - Equipment Engineer - Hamco Division of Kayex Corp., Rochester, NY

ABSTRACT

One of the primary requirements of the DOE/JPL silicon sheet task is to develop a process capable of producing low cost silicon. This silicon must be capable of being processed into solar cells which will yield a solar cell efficiency of 14% AM1.

Kayex completed a contract in March, 1980 (JPL Contract Number 954888) in which the capability of growing 150 kg of 6 inch diameter silicon from one quartz crucible was developed and demonstrated. New technology was generated under this contract which allowed the quartz crucible to be recharged with a new supply of polycrystalline silicon while still under vacuum and at temperatures above the melting point of silicon. However, costs related to the time consuming process of: 1) melting down of the bulk polycrystalline silicon prior to commencement of the crystal grower step and 2) growing of the crystal after completion of the meltdown are the major problems related to the further cost reductions of the crystal growing steps.

The technology generated under this contract was aimed at developing process improvement concepts for lowering the costs of the meltdown and crystal growth functions. A program aimed at improving process automation for increased yield and reduced labor requirement was also undertaken. The development of the various equipment designs that enable high volume, continuous Czochralski production to be achieved should be directly transferable to industry. The specific tasks of the contract were as follows:

1. The design and construction of a modified growth chamber including:
 - a) Specification and provision of a 50 kw high frequency RF generator incorporating a moveable induction coil within the CG2000 RC furnace tank for preheating/melting of polycrystalline rod feed materials at a rate of > 25 kg/hr.
 - b) An induction-heated pre-melter for lump or granular silicon based upon "cold crucible" techniques. The system is to be designed to interface directly into the CG2000 RC furnace tank.
 - c) A cooling coil/radiation shield to accelerate crystal growth, with a crystal growth rate of 15 cm per hour.
2. Provision and demonstration of a microprocessor controlled system for reducing labor cost in terms of hours and skill requirements.
3. Investigation and evaluation of the variations of the effects of the physical form of silicon feed material (rods, chunks, grains) on the crystal growth process and the ultimate crystal produced.

4. Prepare suitable economic cost analysis projection for a low cost, technically feasible prototype growth process.

Following provision and commissioning of the various equipment requirements of the contract, process technology development would commence.

The objectives of the process technology phase was to develop and demonstrate continuous Czochralski crystal growth. Continuous Czochralski growth was defined as a throughput of 150 kg of silicon crystals of 15 cm diameter, utilizing one common crucible with melt replenishment.

The specific objectives were as follows:

- a) Continuous growth of 150 kg or more of multiple single crystal ingots from one common crucible with melt replenishment.
- b) Resistivity of 1 - 3 ohm-cm p type, in all crystals.
- c) Dislocation density below 10^4 per cm^2 .
- d) Diameter of 15 cm or greater.
- e) Growth rate of 15 cm/hr or greater
- f) Orientation: 1-0-0.
- g) Pulled yield of greater than 90%.
- h) Equipment suitable for high volume continuous Czochralski production, transferable directly to industry.
- i) Solar cell efficiency equal to or greater than 14% at AM1.

The conclusions and technology status of the contract as applicable to the objectives of the contract are reported.

Cost projections and actual cost achievements have been developed using SAMICS/IPEG formula and are also reported.

No investigation and evaluation of the variations or the effects of the physical form of silicon feed material on the crystal growth process and impurity build-up in the ultimate crystal produced was undertaken.

1.0 INTRODUCTION

This report presents the conclusions and technology status of the work performed by the Kayex Corporation for the Jet Propulsion Laboratory under contract number 955270 (Tsongas). The contract commenced on March 12, 1979 and was completed on September 30, 1980.

The contract was part of the JPL/DOE Low Cost Solar Array (LSA) project. The goal of the LSA project is to reduce the cost of generating a peak kilowatt of electricity to:

- a) \$2800 per peak kilowatt by 1982 and
- b) \$700 per peak kilowatt by 1986

(both costs expressed in 1980 dollars).

Kayex's efforts on contract number 955270 were aimed at near term cost reduction and yield improvement of a continuous Czochralski crystal growth process capable of producing silicon suitable for production of low cost photovoltaic solar cells.

1.1 Background

Kayex Corporation has been producing Czochralski type crystal growing equipment at its Rochester, New York facility since the late 1960's. The production capacity has been steadily increased through the 1970's to meet the increased semiconductor industry requirements. The market requirements for Kayex crystal growers has established the company as the leading domestic supplier of Czochralski equipment.

As a result of company knowledge of crystal growing furnaces and silicon growth technology, the company was successful in obtaining a DOE/JPL contract (contract number 954888) in late 1977. This contract ran from October, 1977 through March, 1980. It finally resulted in the development of technology which demonstrated continuous crystal growth of 150 kg of 15 cm diameter silicon crystal from one common crucible using melt replenishment techniques.

Whilst contract number 954888 was ongoing, DOE/JPL issued the Kayex Corporation with the present contract (contract number 955270). This contract ran from March 12, 1979 to September 30, 1980.

In September, 1980, a further contract was issued to the Kayex Corporation by DOE/JPL. The contract (contract number 955733) is for development of an advanced Czochralski growth process to produce low cost 150 kg silicon ingots from a single crucible for technology readiness.

1.2 Previously Developed Technology

For the purpose of this report, it is intended that previously developed technology is only briefly described. Comprehensive detailing of the technology

can be found in the final report of DOE/JPL contract number 954888 entitled "Continuous Czochralski Growth".⁽¹⁾

The following is a synopsis of the developed technology:

The technology objective was to demonstrate the growth of at least 100 kg of single crystal ingot from one melt container (crucible) by the Czochralski (CZ) method.

The normal commercial procedure for Czochralski type crystal growth is to grow one crystal from one crucible, replacing the old crucible with a new one each time a crystal is grown.

Although a number of methods of accomplishing continuous CZ growth could have been devised, it was very important that the chosen method would be suitable for a production environment and that it would be attractive in terms of safety, reliability and cost. In a production-type process, variables should be well-controlled and equipment should not require operators with great skill or technical ability. Therefore, Kayex's approach to the continuous growth process relied on conventional CZ technology combined with new equipment designs which allowed repeated alternate cycles of crystal growth and hot melt replenishment by methods which would be suitable for use in a high volume production facility.

The production of several crystals (up to 100 kg total) from one melt container (crucible) by the Czochralski (CZ) method required equipment and process modifications not commonly used in the semiconductor device industry. These modifications were required for the purpose of permitting a method of silicon replenishment without cooling the residual melt and container or contaminating the silicon.

In order to accomplish continuous CZ growth based on the method proposed by Kayex, it was necessary to design a crystal grower with the capability of:

- Continuous high temperature operation of the furnace section and continuous purging with argon at a vacuum of 20 torr
- Isolating the top portion of the crystal grower from the lower furnace section
- Supporting a polycrystalline rod or hopper (lump material) to be used to refill (recharge) the hot crucible before starting another growth cycle.

These three requirements were met by redesigning and modifying a Hamco Model CG2000 crystal grower with a special chamber for the storage of a supply of polycrystalline silicon and a vacuum-tight isolation valve to permit retrieval of crystals and melt replenishment without contamination.

This new grower design, along with low cost objectives for grown silicon, necessitated additional modifications to the new grower and special crystal

handling equipment.

- The newly designed isolation valve necessitated a redesign of the Automatic Diameter Control (ADC) optical system.
- The growth of large crystal ingots required a modified bead chain/cable mechanism.
- The furnace section was redesigned to accommodate a 14" crucible and corresponding hot zone.
- A dopant fixture was designed to allow dopant replenishment to the melt during a recharge cycle without contaminating the melt.
- A special crystal/poly transfer device and a storage rack were designed to handle large, hot crystal ingots.
- A hopper was designed and developed which has successfully demonstrated the recharging of lump poly silicon.

The process for CZ growth of multiple crystal ingots from one crucible proposed by Kayex was dependent on the ability to remove a grown crystal from the grower and hot fill (recharge) the fused quartz crucible without contaminating the melt or allowing the furnace to cool down.

To accomplish the grown crystal removal and subsequent recharging, it was necessary to isolate the furnace section of the crystal grower from the upper portion (pull chamber) of the grower (refer to Figure 1 and Figure 2). In isolating the furnace tank from the pull chamber, a vacuum-tight seal had to be maintained so that the pull chamber could be brought to atmospheric pressure while the furnace tank was still under vacuum. Once the pull chamber was at atmospheric pressure, the door could be opened and the grown crystal removed. A new supply of virgin poly silicon (rod or lumps) could then be placed in the pull chamber, the door closed, the pull chamber purged of air with clean, inert argon and finally pumped down to a vacuum equal to the furnace tank, thus enabling the operator to open the isolation valve and proceed with the recharge cycle. Separate vacuum systems for the furnace tank and pull chamber were necessary to accomplish this cycle. A flow diagram of a continuous CZ cycle is included in Figure 3, page

To meet the economic goals of the project, it was felt that time and cost savings would be accomplished if a supply of polysilicon material (rod or lump) could be contained in the pull chamber while a crystal was being grown. This would allow the operator to recharge the crucible, then grow the next crystal, eliminating one cycle of opening the pull chamber door. Therefore, a polyweight Recharge System was designed with its own cable system to hold a supply of polysilicon material for recharging.

The newly designed isolation valve was not compatible with the old automatic

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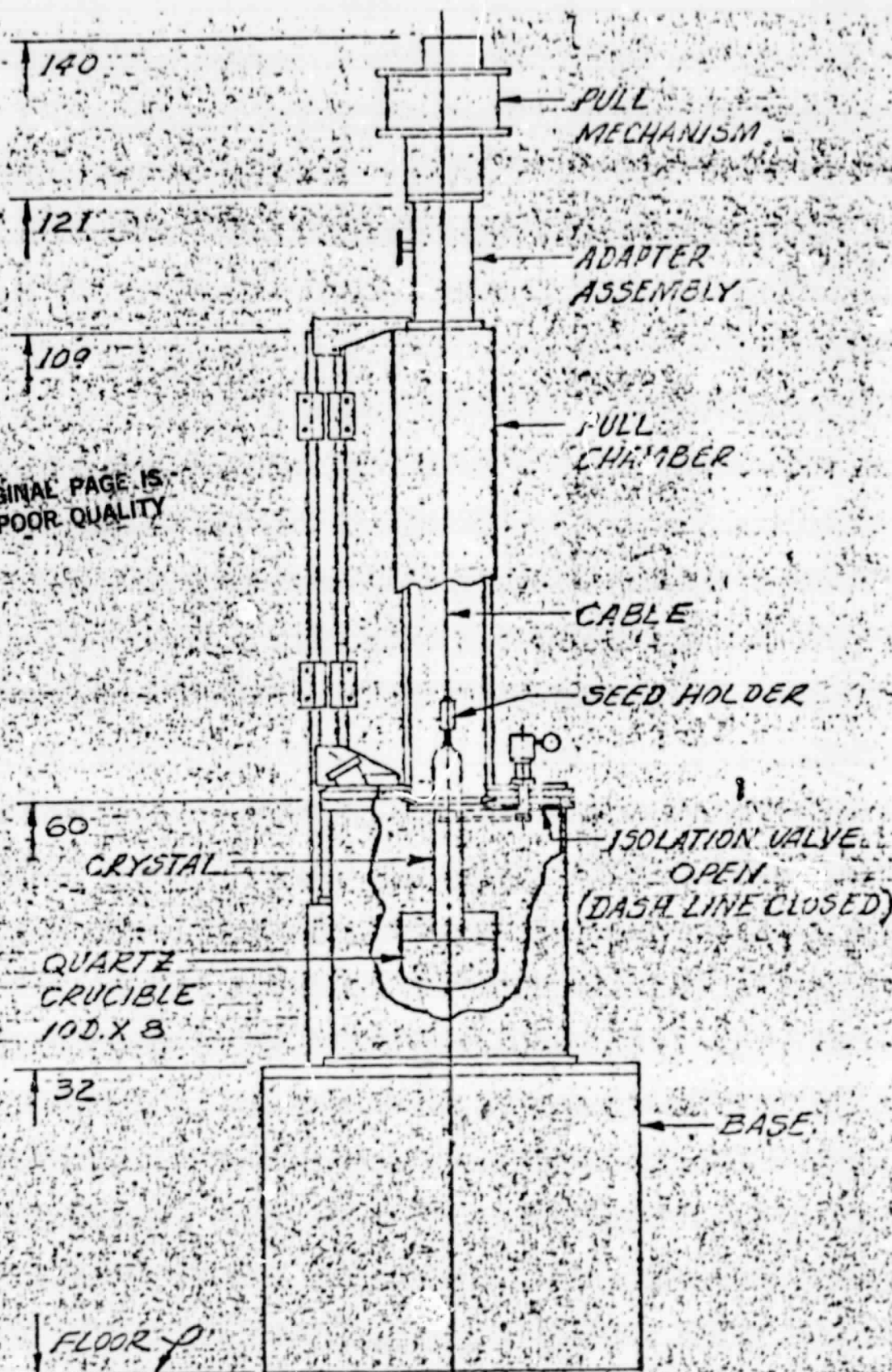
TOLERANCES MACHINE

SYM
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HAMCO DIV. OF THE KAYEX CO
ROCHESTER, N. Y.

DESCRIPTION OF CHANGE

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FRONT VIEW
SINGLE CZOCHRALSKI
GROWTH FACILITY

Figure 1

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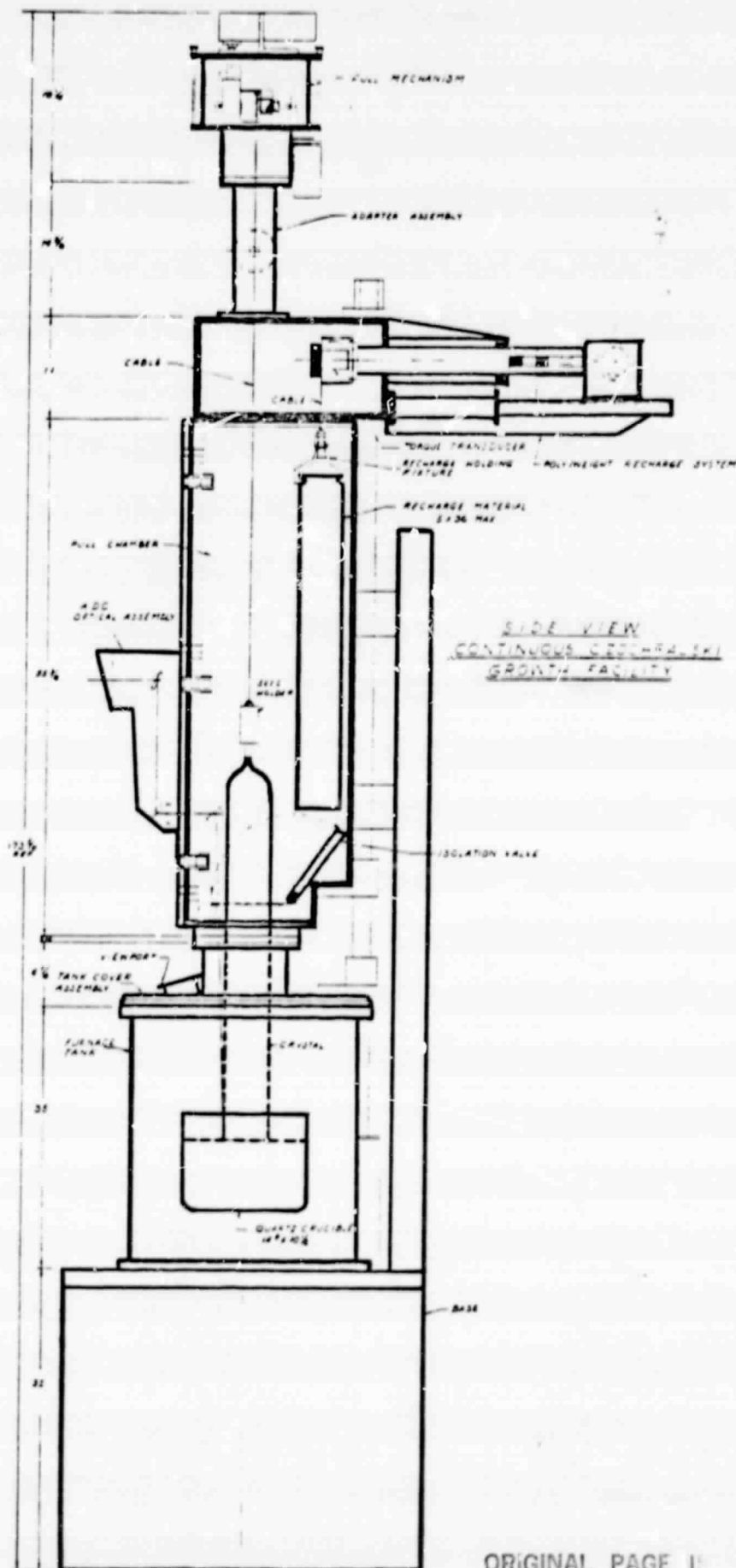
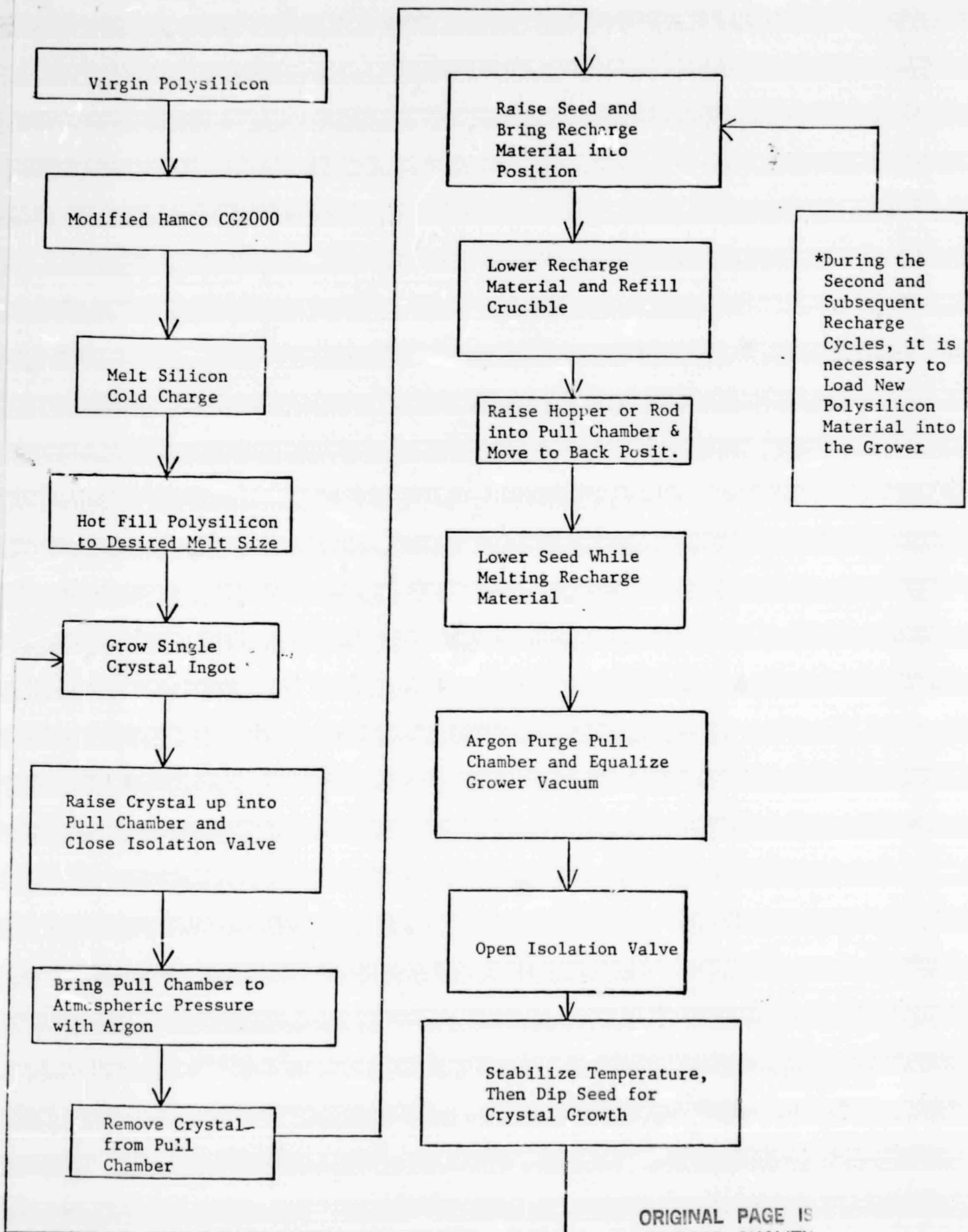


Figure 2
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FLOW CHART FOR CONTINUOUS CZ GROWTH

Figure 3



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NOTE: TO ALTER FOR INDIVIDUAL ARMING ADJUSTMENT:

1. ADJUSTMENT OF SLACK IN CABLE

2. ADJUSTMENT OF SLACK IN CABLE

3. ADJUSTMENT OF SLACK IN CABLE

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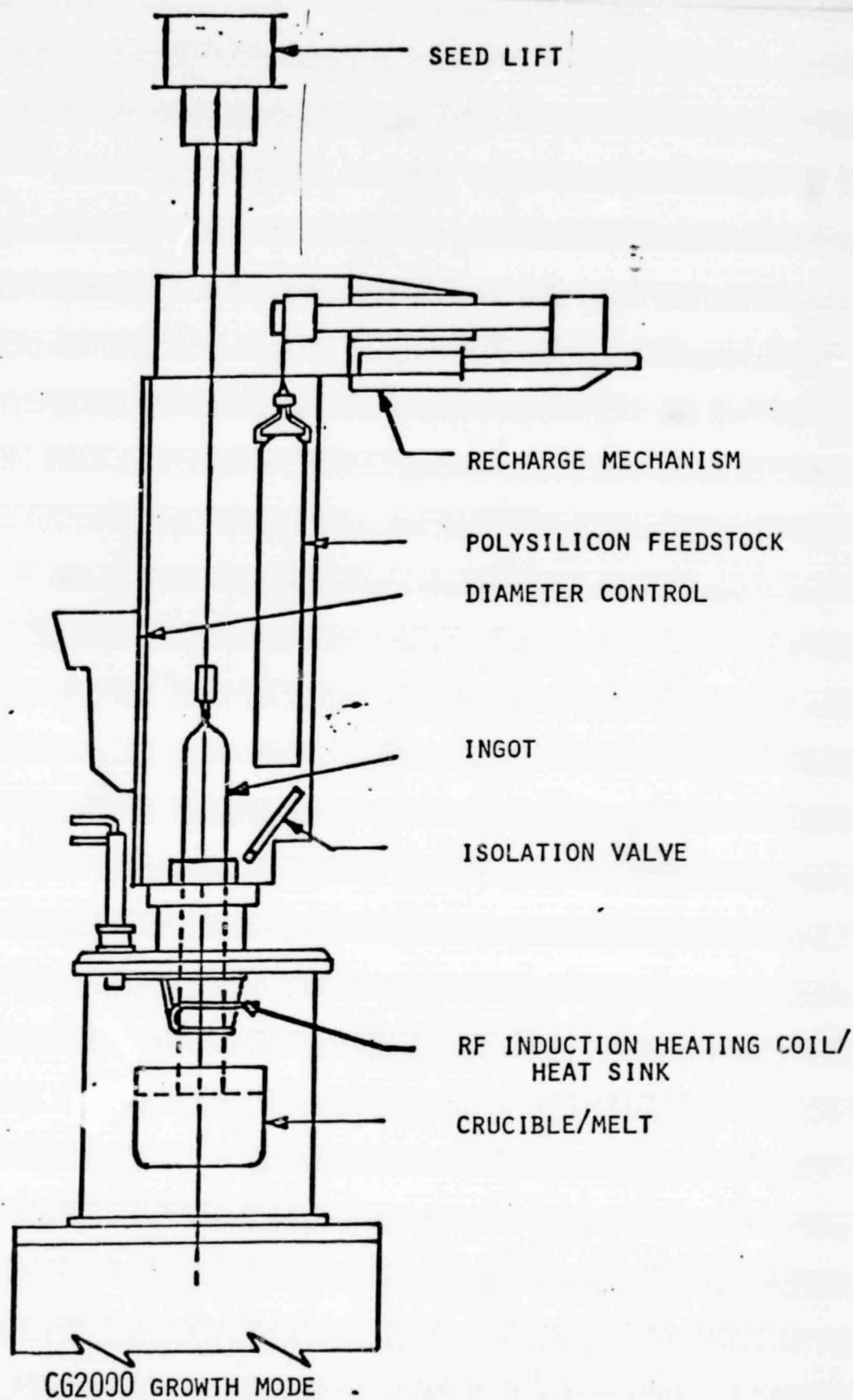
25. ADJUSTMENT OF SLACK IN CABLE

26. ADJUSTMENT OF SLACK IN CABLE

27. ADJUSTMENT OF SLACK IN CABLE

Part No.	Description	Quantity	Notes
1	Base	1	
2	Boom	1	
3	Hydraulic Cylinder	2	
4	Hydraulic Hose	2	
5	Hydraulic Valve	1	
6	Hydraulic Filter	1	
7	Hydraulic Pump	1	
8	Hydraulic Motor	1	
9	Hydraulic Switch	1	
10	Hydraulic Control	1	
11	Hydraulic Line	1	
12	Hydraulic Fitting	1	
13	Hydraulic Seal	1	
14	Hydraulic Gasket	1	
15	Hydraulic Bolt	1	
16	Hydraulic Nut	1	
17	Hydraulic Washer	1	
18	Hydraulic Lock	1	
19	Hydraulic Pin	1	
20	Hydraulic Ring	1	
21	Hydraulic Collar	1	
22	Hydraulic Flange	1	
23	Hydraulic Bracket	1	
24	Hydraulic Support	1	
25	Hydraulic Base	1	
26	Hydraulic Mount	1	
27	Hydraulic Anchor	1	

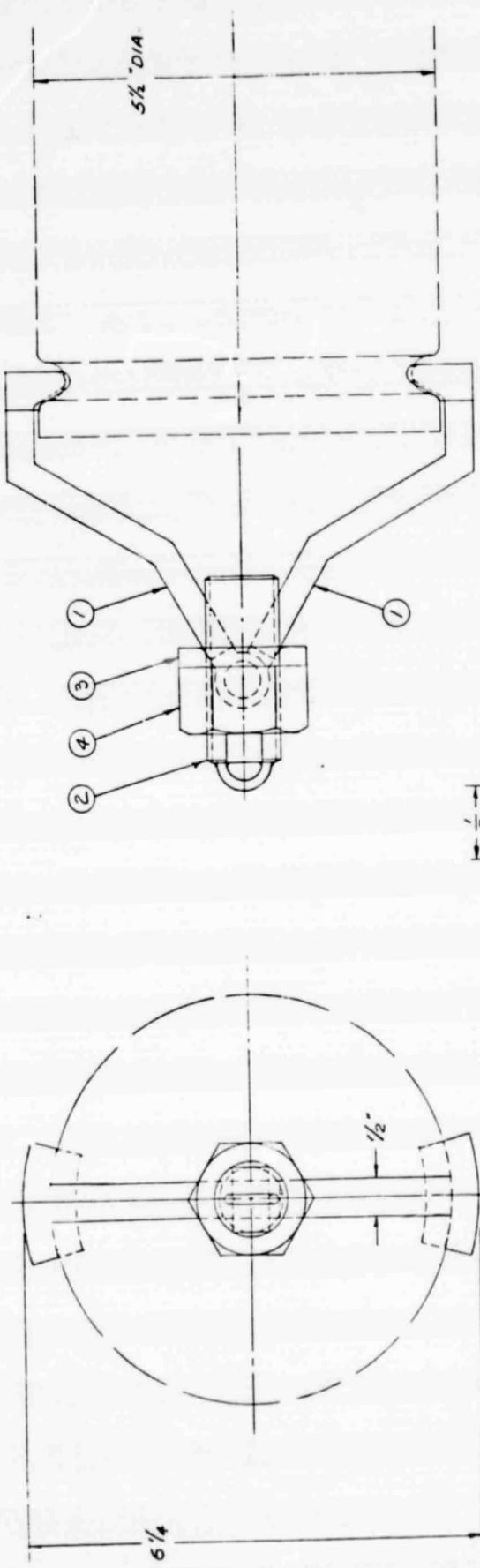
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Modified Growth Chamber Showing Stored Poly Feed Rod

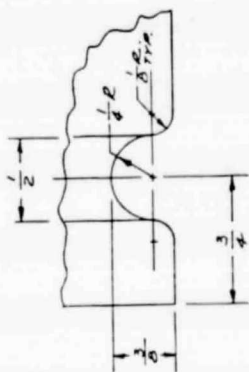
Figure 5

REV	DATE	DESCRIPTION	PART NO.	QTY
1	021-0065-201	HOOK-PC HOLDING FEATURE	2	
2	024-0079-201	ROD-PC HOLDING FEATURE	1	
3	027-0080-101	WASHER-PC HOLDING FEATURE	1	
4	026-0001-43B	NUT-1"-B STD 3.5.	1	



SIDE VIEW

PLAN VIEW



GROOVE DETAIL FOR ALL
RECHARGE MATERIAL
SCALE: 2 X

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POLY ATTACHMENT DEVICE

FIGURE 6

REV		DATE		DESCRIPTION	
1		021-0065-201		HOOK-PC HOLDING FEATURE	
2		024-0079-201		ROD-PC HOLDING FEATURE	
3		027-0080-101		WASHER-PC HOLDING FEATURE	
4		026-0001-43B		NUT-1"-B STD 3.5.	

REV		DATE		DESCRIPTION	
1		021-0065-201		HOOK-PC HOLDING FEATURE	
2		024-0079-201		ROD-PC HOLDING FEATURE	
3		027-0080-101		WASHER-PC HOLDING FEATURE	
4		026-0001-43B		NUT-1"-B STD 3.5.	

REV		DATE		DESCRIPTION	
1		021-0065-201		HOOK-PC HOLDING FEATURE	
2		024-0079-201		ROD-PC HOLDING FEATURE	
3		027-0080-101		WASHER-PC HOLDING FEATURE	
4		026-0001-43B		NUT-1"-B STD 3.5.	

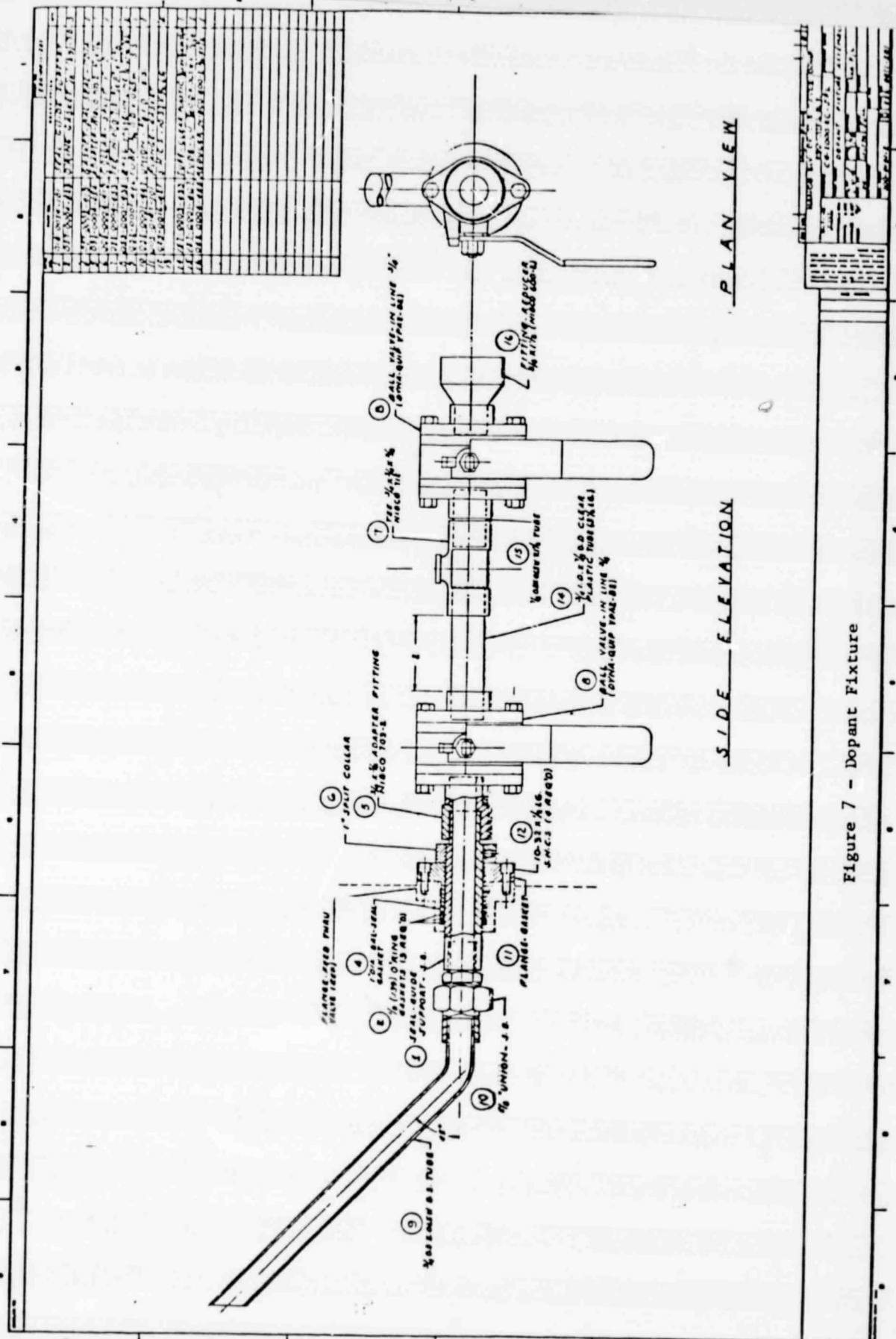


Figure 7 - Dopant Fixture

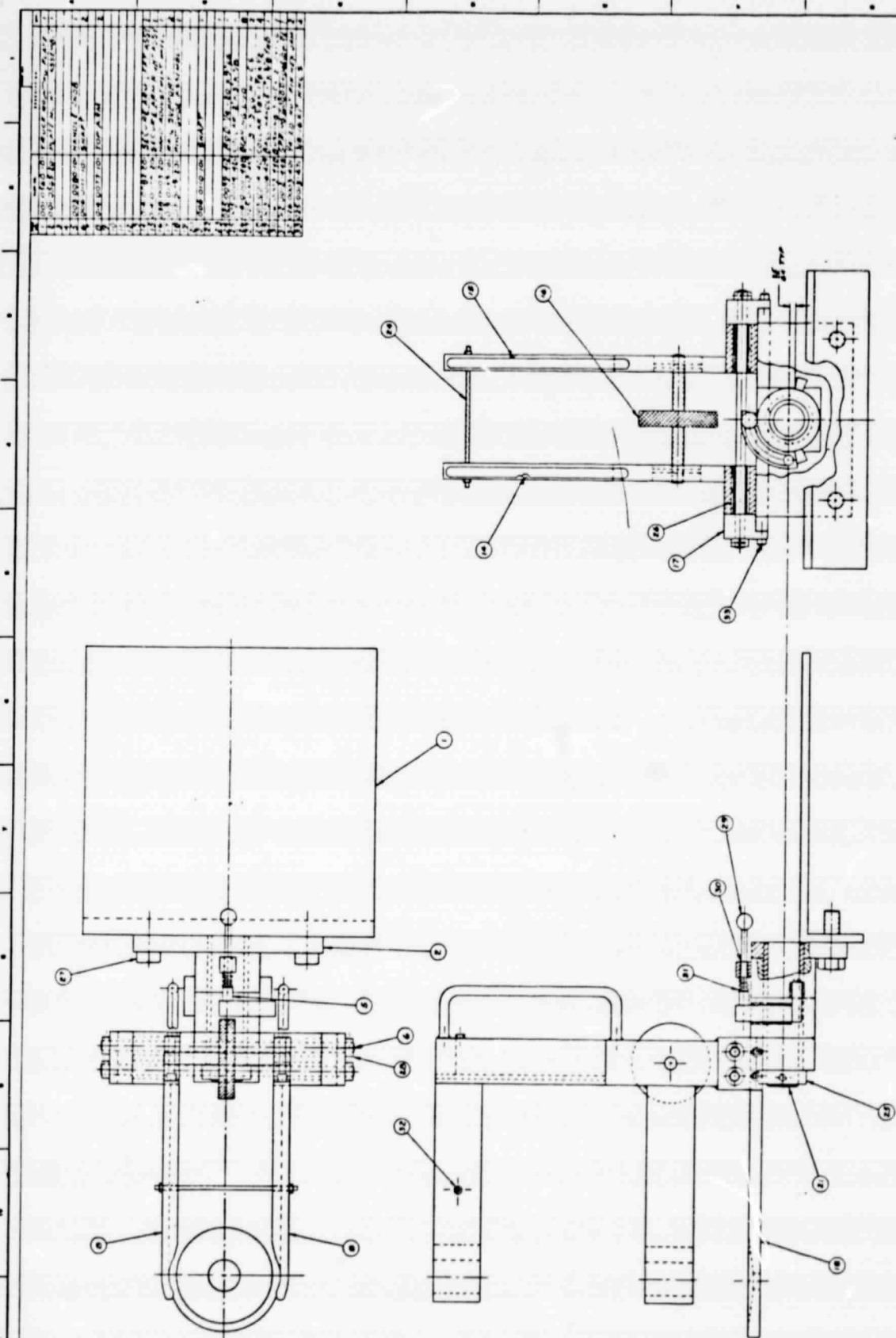
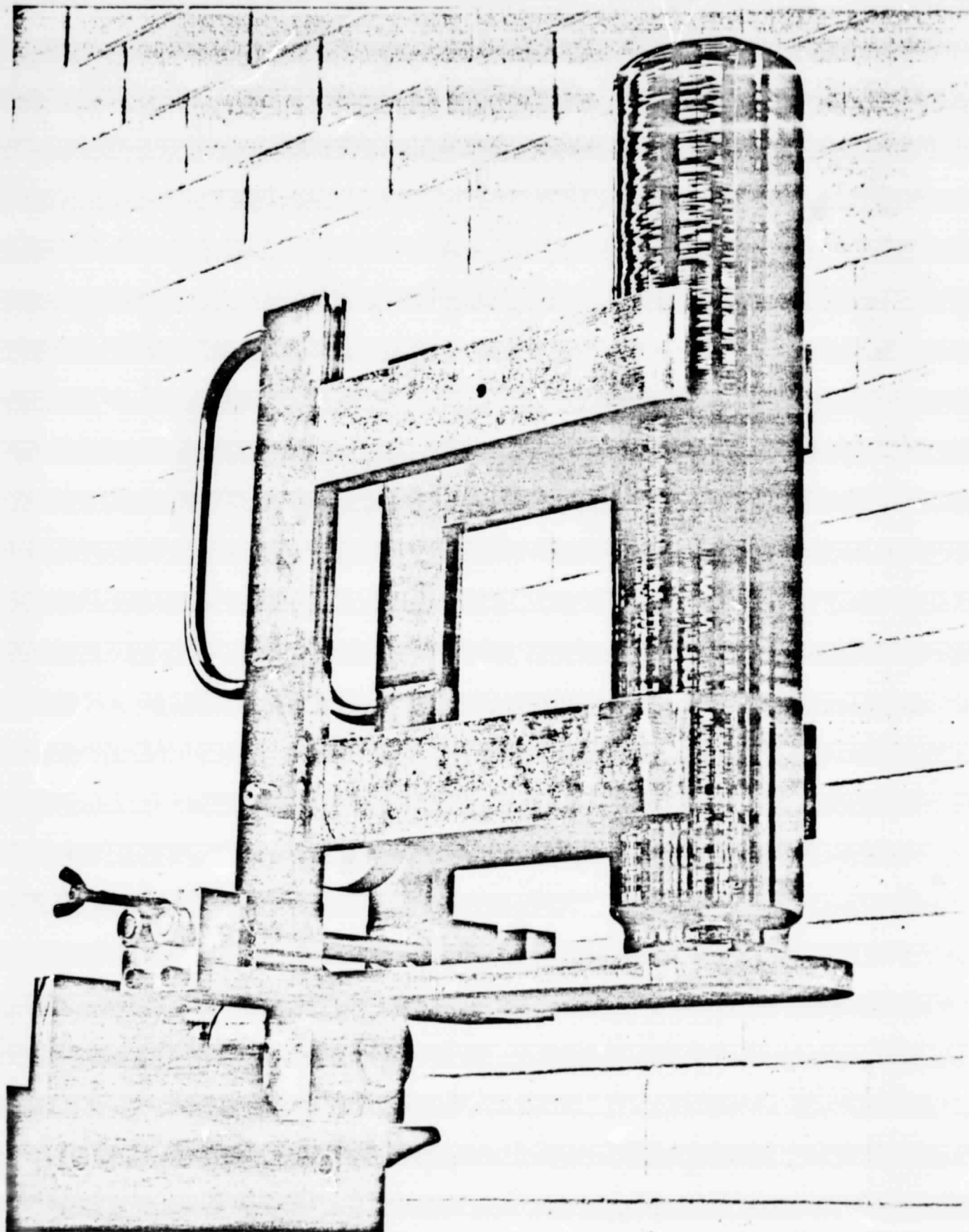


Figure 8 - Crystal/Poly Transfer Device



CRYSTAL/POLY TRANSFER DEVICE

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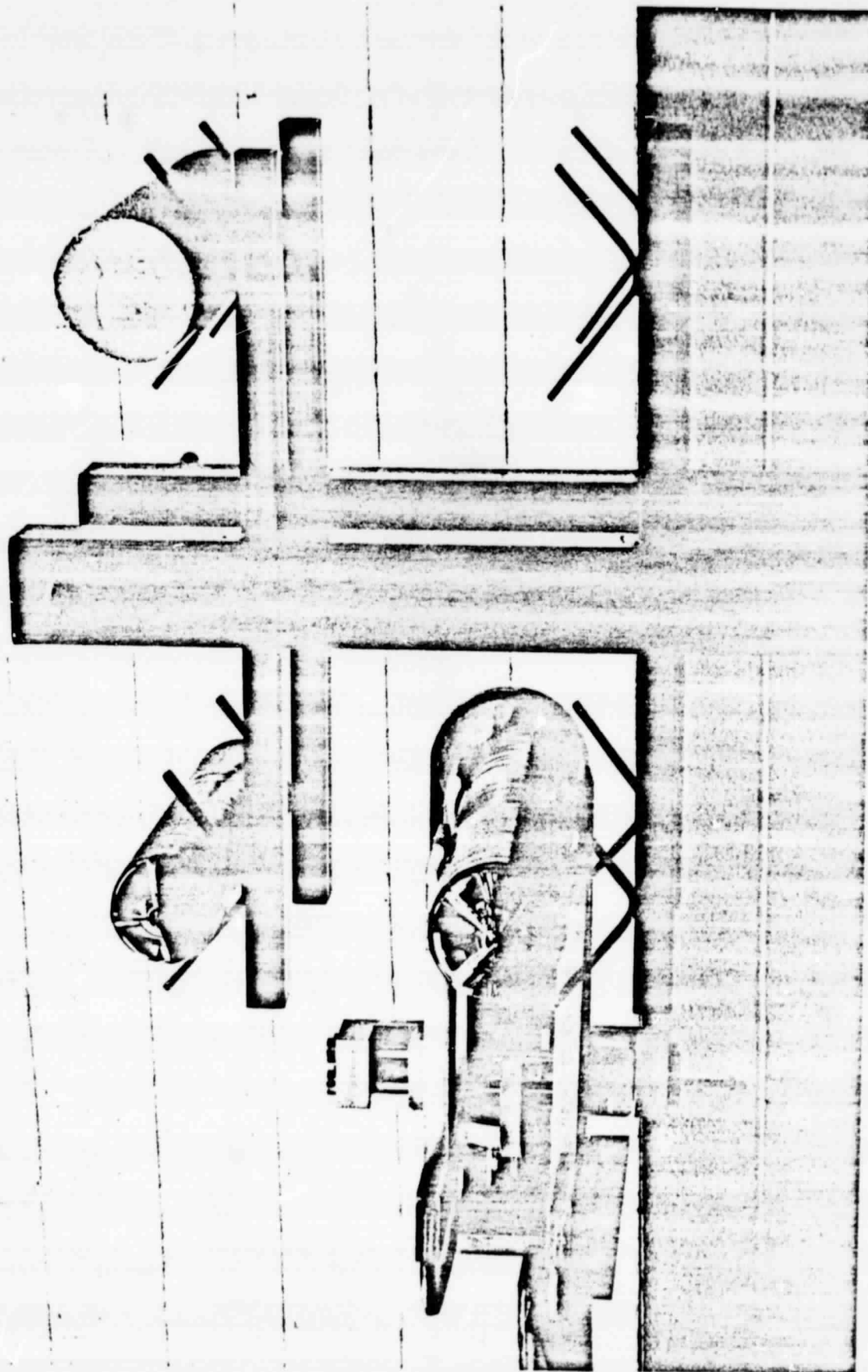
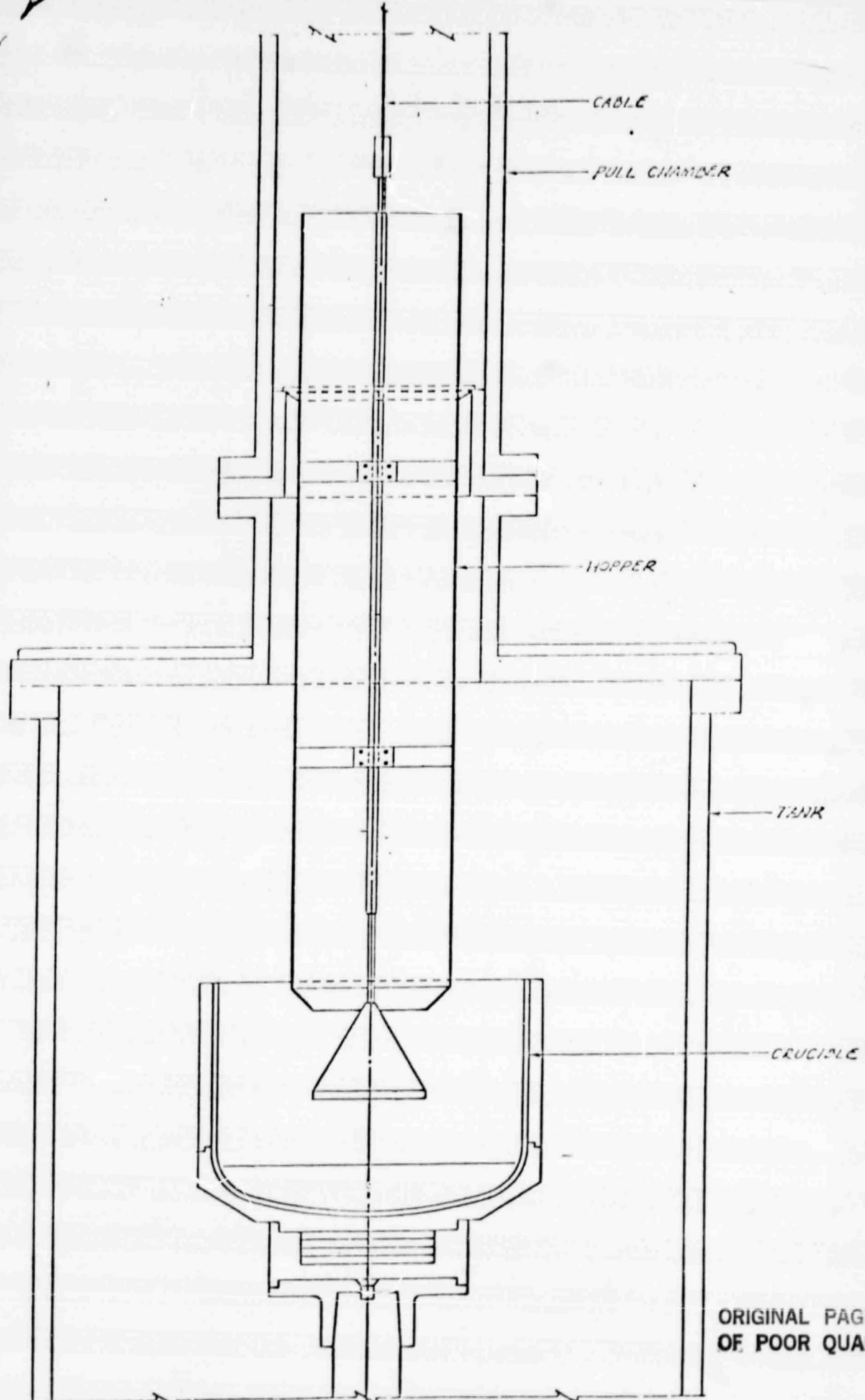


FIGURE 13 PLACING HOT CRYSTAL ON COOLING RACK



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Lump Silicon Recharging Hopper
Figure 11

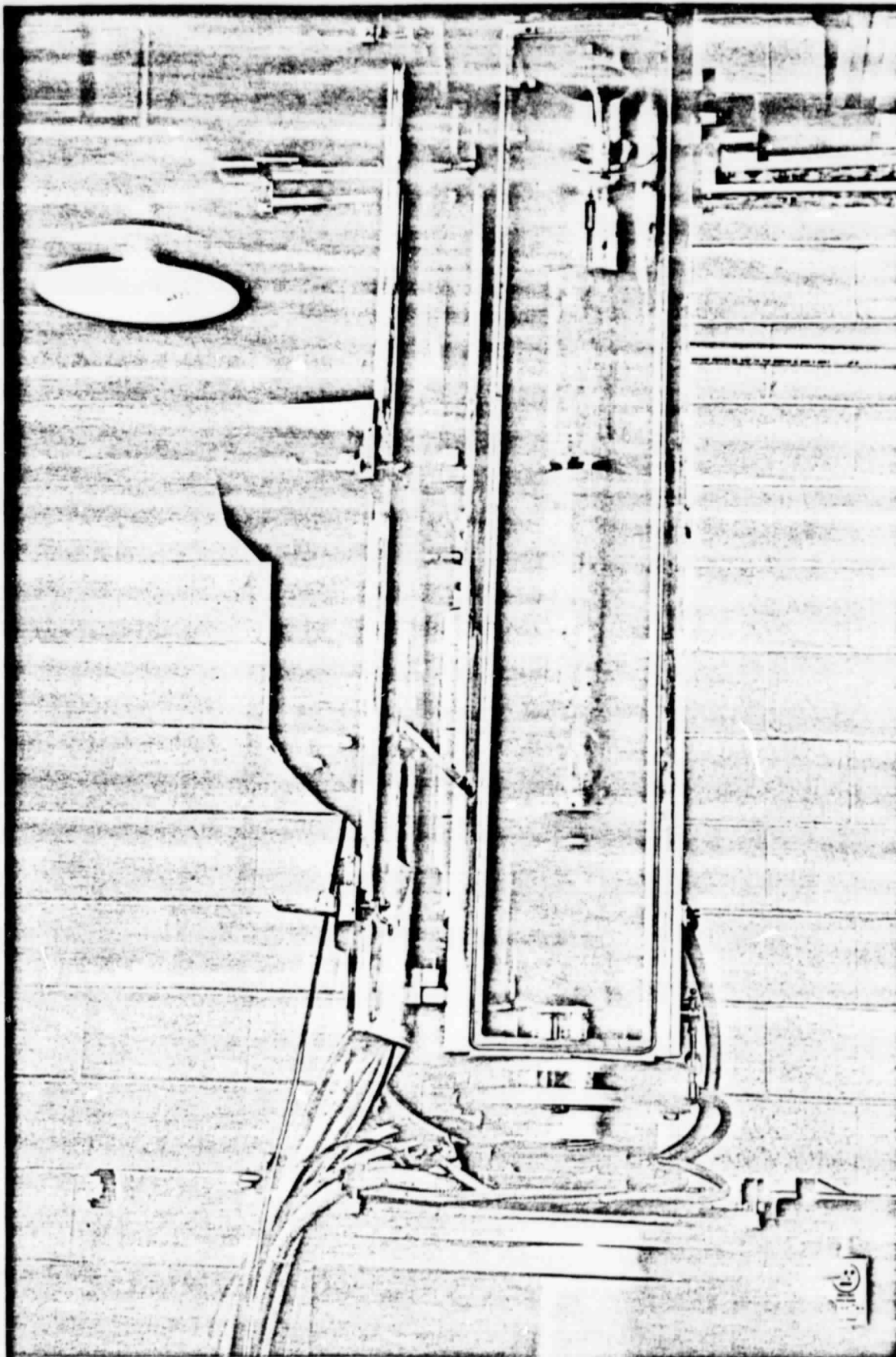


Figure 12
Lump Silicon Recharge Hopper. Shown in Growth Chamber

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diameter control optical system (ADC). An ADC optical system utilizing appropriate lenses for sighting through the valve throat was designed and built for the new grower (Figure 4). It was also necessary to see the new flapper type isolation valve relative to the grown crystal and polysilicon material to be used for recharging. Therefore, a larger viewport window was designed into the pull chamber door for this purpose. The pull chamber had to be modified further (enlarged) to provide space for storage of polysilicon (Figure 5).

The growth of large crystals (4" diameter) from large melts was highly cost effective and was included as part of the 954888 contract proposal. This objective could be accomplished by increasing the capacity of the melt container (crucible). A 14 inch diameter crucible was calculated to be the optimum size for the project goals. However, a newly designed 14 inch hot zone required a larger furnace tank than the standard CG2000 equipment. The newly designed furnace tank was capable of accommodating either the 12 inch or 14 inch hot zone. With the increase in melt size and the corresponding size and weight of the crystals grown, the need for a stronger bead chain seed lift device became necessary. The seed lift pull mechanism was modified to utilize a stainless steel cable. Mechanical and electrical modifications were also included to permit weight measurement of ingots up to 50 kilograms.

Two problems had to be solved when it was decided to recharge using polysilicon rods:

- 1) A method of attaching the rods to the support cable had to be developed.
- 2) A method of adding dopant to the crucible while it was hot and under vacuum required development.

The first problem was solved by designing a poly attaching device (Figure 6). This clamp type of design required notching of the poly silicon rod. The second problem was solved by designing a dopant fixture (Figure 7) that would allow for insertion of dopant pellets, incorporation of a vacuum interlock system and rotating capabilities such that it could be positioned over the crucible (melt). The dopant pellets could then be released and the fixture rotated out of the way so that the next crystal could be grown. The dopant fixture was designed to fit into the furnace tank cover.

Finally, since the continuous CZ process did not allow for the prolonged cooling of grown crystals in the crystal grower, it was necessary to design and build special crystal handling and storage devices (Figures 8 and 9) which could accommodate large, hot crystals and large poly silicon rods. Figure 10 shows a crystal being held by the crystal/poly transfer device ready to be placed into the crystal/poly storage rack.

A major design and process study was necessary to recharge lump poly silicon. A lump recharging system was developed incorporating a self-dumping hopper design (Figures 5, 11, and 12). The hopper used during the 954888 project allowed for a maximum recharge capability of 18 kilograms of poly silicon lumps.

2.0 PROCESS TECHNOLOGY

The Kayex Corporation had already been granted a DOE/JPL contract (954888) which was aimed at the development of a "continuous growth" process for manufacturing silicon by the Czochralski method. "Continuous growth" is described as the manufacture of over 100 kg of ingot from one crucible by recharging of the crucible.

To date, two methods of recharging had been developed:

- a) melting of a polycrystalline billet of silicon by lowering the billet into the melt in a slow and controlled manner,
- b) using a specially designed storage hopper, filled with screened silicon chunk material. The hopper is lowered to a predetermined point above the crucible. The self-dumping action of the hopper system is then automatically activated, allowing the hopper to empty.

Both of these systems had been developed together with the poly rod weight recharge system. This system allowed for alternate storage/melting or recharging of material for re-filling the crucible.

During crucible recharging using either of these techniques, it is necessary to increase the furnace temperature approximately 250 degrees Centigrade above the melting point of silicon. This is to ensure a reasonable melting time for the recharged material. However, this does impact on both the cost and the process due to:

- a) cost of the increased power requirements
- b) effect of devitrification of the crucible due to the thermal cycling of the crucible.

Further cost reductions were aimed for by utilization of a suitable heat sink arrangement in the crystal puller. The heat sink was used to accelerate the growth rate by effective removal of the energy generated by the heat of fusion.

The final objective was to automate the growth process by development of microprocessor control. The purpose of this objective was the reduction of operator error and so improve yield and cost effectiveness.

The various programs developed under this contract were divided into the following definitive parts:

- a) Recharging of the crucible utilizing polycrystalline silicon rod feedstock and melting these rods directly into the crucible using RF induction heating techniques.
- b) Development of microprocessor control for Czochralski crystal growth.
- c) Utilization of the RF induction heated water-cooled coil as the heat sink to accelerate the crystal growth process.
- d) Recharging of the crucible by pouring molten silicon directly into the crucible using cold crucible/silver boat levitation techniques.

Each of the programs will be reported separately and in detail as follows:

- a) Accelerated melt replenishment
- b) Microprocessor control
- c) Accelerated crystal growth
- d) Cold crucible melting.

RF Generator Selection and Polycrystalline Feedstock Selection

Several RF generator vendors were asked to submit quotations for the supply of a suitable RF generator to satisfy process requirements. Cycle Dyne (Jamaica, NY) offered the use of laboratory test facilities in order that we could determine the power requirements necessary for melting up to 5" diameter polycrystalline rod. These trials were undertaken on May 16, 1979.

From the trials, it was determined that a 450 KHz, 50 kw output generator was required. Trials were completed using both a multi-turn high voltage coil and a low voltage single turn coil. Both systems had the ability to melt a 5" diameter polycrystalline rod.

Based on this fact, a report was submitted to JPL for consideration and authorization to purchase either a Lepel or a Cycle Dyne generator. The authorization was granted based on our recommendation to purchase Cycle Dyne. The order was placed for delivery on August 20, 1979.

The major consideration for selecting Cycle Dyne was the versatility of the equipment. Both the single turn coil system and the multi-turn coil system could be remotely fed from the generator via separate remote stations. The remote stations could be fed by co-axial cable from the generator and then to the crystal puller. The design package therefore allowed the single turn coil to be utilized for the accelerated melt program and the multi-turn coil to be utilized for the cold crucible program. Also, as the single turn coil would be designed such that it was adjustable through the vertical plane, it was planned to use it as the heat sink in the accelerated growth program. By utilizing these remote station systems, it would allow work to be done on either program without having to shut down for the equipment to be transferred from one system to the other. Cycle Dyne was also slightly more price advantageous over Lepel.

Quotations were requested for the purchase of suitable quality silicon poly rods. Replies were received from Dow Corning and Montedison USA. Dow quoted a price of \$86.55 per kilo for either 4" or 5" diameter material. The major consideration in the purchase of poly rod for this project was one of availability and guarantee of being "crack free". Dow would only guarantee up to 3" diameter poly rod as "crack free". The ability therefore of being able to purchase "crack free" feedstock was considered paramount, both from achieving the thruput aspects of the program and from a safety viewpoint.

Montedison USA quoted a price of \$70 per kilo. This was price advantageous over Hemlock Semiconductor Corporation by \$16.65 per kilo. The material was nominally 5" diameter and, although an initial request was made for "crack free" material, suppliers could not guarantee it to be so. Material was therefore supplied on a "best effort" basis.

2.1 Accelerated Melt Replenishment

Meltback of Polycrystalline Rod Using an RF Coil

Polycrystalline silicon rod would be melted using an RF induction heated copper work coil. The rod would be stored and alternately melted in order to recharge the crucible after completion of the pulling cycle.

Problems encountered during the design and fabrication stages were:

1. Coil configuration and matching of the coil to the RF feed-thru.
2. Design and construction of the RF feed-thru.

Discussion:

A single turn machined copper fabricated work coil was utilized for the accelerated melt program. Power was fed to the coil by means of a 50 kw RF induction heated generator operating at a frequency of 450 KHz. The power was fed by co-axial cable to a remote switching box located on the puller framework. Power was then fed from the remote box via a specially constructed RF feed-thru system sited in the spectator viewport of the puller. Power was then fed from the generator to the work coil via the remote box and feed-thru.

The work coil was fabricated from machined copper, but required some modification to allow it to be connected to the RF feed-thru. By fabricating from machined copper, the wall thickness was thicker than conventional tubing. The coil was water-cooled; the increased wall thickness therefore allowed the coil more protection against fracture should arcing occur.

The coil was cone-shaped with an ID of nominally 6.5". The cone was angled at 30° to the top wall ID with a total band width of approximately 4". The coil therefore had the capability of melting and pulling of crystals of up to 6" diameter. Melting trials indicated that, by having a slightly cone shaped coil, the melt could be effectively "squeezed", such that a pouring effect of the melt could be achieved.

Major problems were encountered in locating a subcontractor who had the capability and the willingness to fabricate the RF feed-thru. The one contractor who originally agreed to build finally returned the drawings after one month and declined to build. They stated that they could not guarantee the fabrication because of the anticipated high heat load. The feed-thru was therefore fabricated in-house.

Extreme difficulties were experienced in locating material suitable for fabrication, i.e. machinable copper. The system is illustrated in Figure 13.

These delays resulted in a delay in the commencement of the accelerated melt program. However, it did not prevent the standard products testing department from pulling a crystal from a 17 kilogram silicon charge using the equipment in its standard form. This was followed by a 100 kilogram run as part of JPL Contract #954888 program. This was requested by Dr. Kachare, JPL Project Monitor. These runs allowed the RF feedthru and work coil installation to be completed.

All initial work was undertaken using 12" purified graphite piece parts and pulling 5" diameter crystals. When the accelerated melt parameters had been established, 14" purified graphite piece parts were installed and 6" diameter crystals were pulled as required by the program.

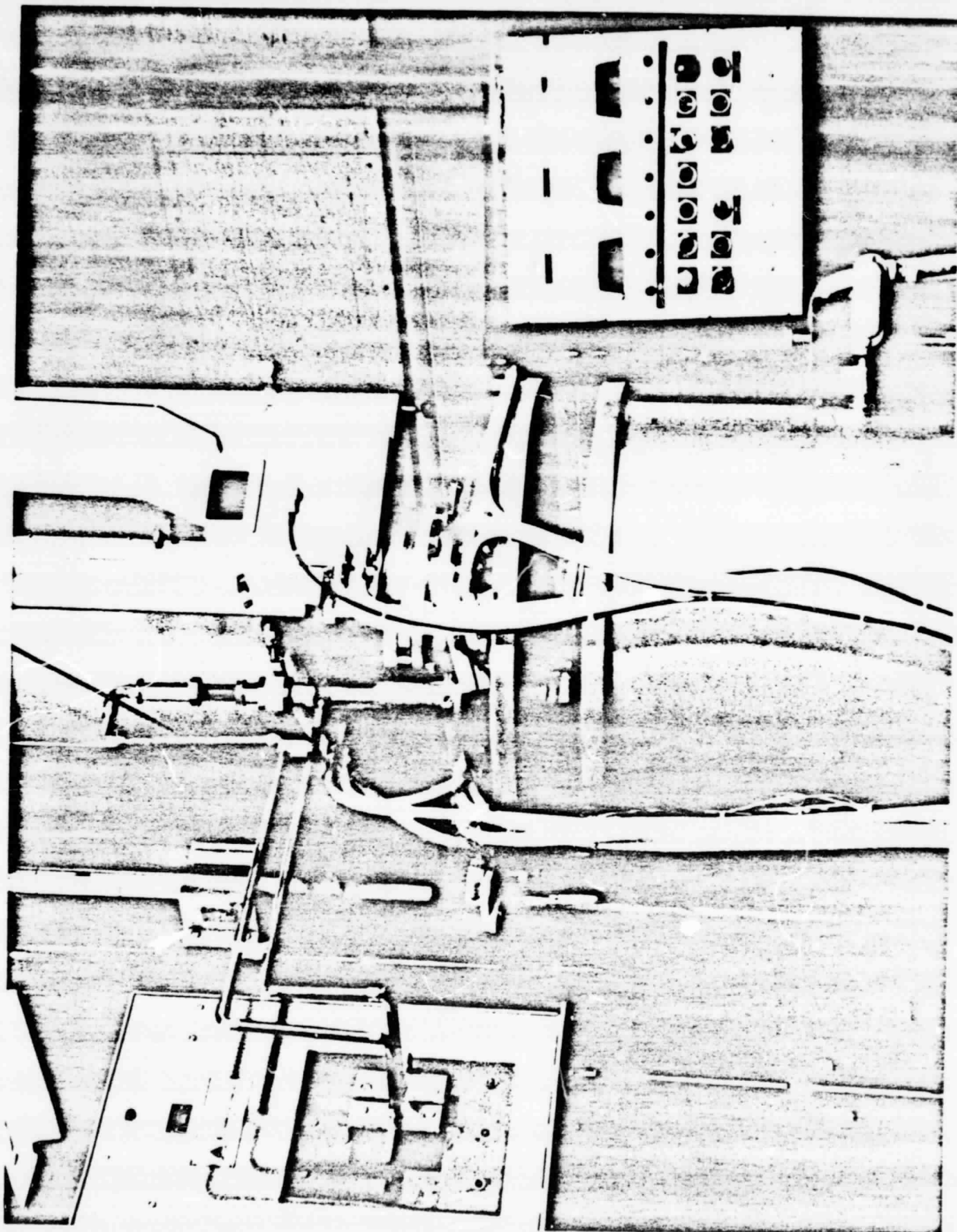
A combination of problems became evident during the commissioning stage of the RF poly rod melting program. Initially, insufficient power transfer and overheating of the RF feedthru system required correction. The power transference problem was overcome by reworking of the power transmission lines for the feedthru.

The two connections into the feedthru were modified such that they entered the feedthru in parallel to ensure that efficiency losses were minimized.

A secondary and ongoing problem was associated with overheating and breakdown of the RF feedthru. Initial overheating was due to RF coupling to a stainless steel section in the feedthru and was overcome by substitution of the stainless steel section with teflon. Voltage breakdown of the teflon sheet insulation within the feedthru was partially overcome by substituting the teflon sheet with a quartz tube.

Melting of polycrystalline rod was demonstrated in atmosphere following the correction of the power transfer problem and partial correction of the feedthru overheating problems. Melting within the CG2000 RC crystal grower was then attempted under normal growth atmosphere conditions, i.e. vacuum/argon pressure at 20 torr. The polycrystalline rod was lowered slowly into the crucible which was being heated by the graphite resistance heater. The temperature of the poly rod was raised sufficiently to allow the poly rod to conduct, then the RF system was switched on to the work coil. The poly rod was raised into the coil and the power increased to attempt melting.

Arcing immediately occurred as the power was increased, primarily as ionization of the argon fill gas. Arcing also occurred around areas of the coil ID and around the insulation between the coil arms. All the sharp edges of the work coil and coil arms were "rounded off" and polished and the melting experiment repeated with the same results. Some arcing between the jaws of the poly rod support chuck and the poly rod was visible. This was corrected by fitting an insulating section in the



RF FEED THROUGH SYSTEM

Figure 13
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cable supporting the poly rod and chuck.

Melting was then attempted utilizing helium as a substitute for argon as the fill gas. Arcing occurred as previously.

Melting was then attempted under argon at atmospheric pressure. Initial melting of the poly rod commenced before ionization of the argon atmosphere occurred within the furnace tank and voltage breakdown also occurred within the RF feedthru insulation. This resulted in fracturing of the quartz insulation within the feedthru and the arms of the RF work coil. Severe cracking of the poly rod was also evident as the power was increased. The cracked section remained intact, however, and fortunately did not fall into the quartz crucible. All of the poly rods also exhibited some degree of "bow" along their vertical axis. This "bow" problem created problems in centering of the poly rod within the work coil. Arcing appeared to be induced in the area of the coil nearest to the poly rod; also, non-symmetrical melting occurred due to the coupling variance within the coil. Operation of the system at atmospheric pressure caused considerable oxide build-up on the work coil and the furnace tank and pull chamber walls.

After replacement of the feedthru insulation and removal of the carbonization on the work coil, a further attempt to melt was made. This attempt was made using helium at atmospheric pressure. It resulted in full generator output being achieved and maintained for approximately 15 minutes without either ionization or multipactor discharge of the growth atmosphere occurring. Considerable melting of the poly rod was achieved (approximately 8 kilograms) before severe arcing occurred within the feedthru assembly resulting with fracturing of the quartz insulator.

An analysis of the RF accelerated melt equipment indicated that:

1. A re-design of the RF feedthru system was necessary to ensure that the electrical failures due to arcing and resultant breakdown of the assembly be eliminated.
2. Ionization and multipactor discharges in both the argon and helium gasses at 20 torr pressure existed.
3. Optimization of the impedance match of the feedthru and the poly rod was critical.
4. Arcing between the poly rod and the copper work coil could introduce copper into the chamber atmosphere in vapor form.

The main effort relating to the re-design of the RF feedthru would be related to improving the durability and reliability of the system. Improvements were considered necessary to reduce the electric field gradients and to improve the insulator design to prevent internal breakdown. The present feedthru suggested that a large percentage of the RF system was used up in arcing. Elimination of this loss would improve the system's efficiency and power transference capability.

Ionization of both argon and helium fill gasses within the furnace tank were probably due to voltage breakdown of the furnace tank atmosphere. At atmospheric pressure and above, the breakdown voltage increases linearly with pressure. At some low pressure, a minimum breakdown potential occurs. At lower pressures, the breakdown voltage rises rapidly. The optimum point is frequently in the 0.1 mm Hg region. The furnace tank was operating near this level and could have been contributing to the ionization problem.

Problems appeared to exist in relation to the impedance match of the RF feedthru and the poly rod.

Measurement of the feedthru minus the poly rod indicated an inductance of approximately 1.26 μ Hy, which presented a small impedance of $-j3.56$ ohms at 450 KHz. The resistance loss in the work coil was only a few milliohms. When a conductive poly rod is placed in the coil, the inductance will fall and the resistance will rise. If the resistive component rose as high as 1 ohm, the current in the coil would be approximately 220 amps if the full 50 kw was transferred to the poly rod. The work coil voltage drop would then be approximately 600 volts. The amount of arcing and overheating of our system at much lower power levels was not in keeping with such voltages. We suspected that higher frequencies were being generated giving rise to higher voltages.

Further evaluation and modification of the RF feedthru was necessary to achieve optimum melting conditions.

The redesigned poly rod recharge mechanism was received by the subcontractor. Problems existed with the drive shaft sealing system and the total system was rejected. The shaft seal arrangement was modified and the system returned. The system was leak checked prior to installation and several leaks were located. The plate arrangement required rewelding prior to installation of the system on the puller. The strain gauge in the poly rod weight system developed a fault and was replaced. The system was installed and calibrated.

As a result of the JPL issuance of the technical direction memorandum in April, 1980, the above program was de-emphasized and no further work was undertaken during the remainder of the contract.

As previously stated, polycrystalline silicon rods of 5-inch diameter guaranteed "crack free" were unavailable. Also, rods exhibiting uniform diameter and free of bow were not obtainable. Diameter variances and excessive bowing down the rod axis created severe melting and arcing problems. The cost of polycrystalline silicon rods also made it highly improbable that the overall cost goals could be met. No known programs are ongoing to produce low cost rods of suitable quality for this program.

A SAMICS/IPEG cost analysis, detailing CZ add on cost per kilogram, using polycrystalline silicon rods as feedstock material, is included in the Economic Analysis

section.

As a result of JPL issuance of a technical direction memorandum in April, 1980, the priorities of the project were redirected as follows:

Priority 1 - development and demonstration of microprocessor control

Priority 2 - accelerated growth program

Priority 3 - development and demonstration of cold crucible melting and crucible recharging concepts

Priority 4 - RF melting of polycrystalline feed rods as a method of recharging the quartz crucible.

2.2 Microprocessor Controls

The purpose of the microprocessor control development program was twofold:

- a) reduced cost
- b) improved yield.

Continuous crystal growth operations necessitate the use of skilled operators to both monitor and operate crystal growers. Typical machine/operator ratio is 3:1. Conservative IPEG calculation for continuous CZ growth of 4" diameter crystal indicates that labor accounts for approximately 17.5% of the total CZ add on cost. By development of a suitable microprocessor control system, it is felt that the crystal grower:operator ratio could be improved to 6:1, thus presenting a substantial saving in labor cost.

Standard operation of a Czochralski crystal grower requires substantial operator effort prior to the operator being able to switch the straight growth cycle into an automatic crystal growth mode. The operator is required to: load, unload and clean down growers, manually melt and temperature stabilize the charge, dip the seed and pull the "fast neck" taper and shoulder the crystal at the appropriate diameter as well as making the necessary pull rate and temperature adjustments to maintain the necessary growth parameters and crystal structure requirements. Calculation and adjustment of crucible lift ratios are also required. Development of crucible recharging techniques have created additional responsibilities on the operator, i.e. crystal retrieval and crucible recharging operations. It should be noted that in a production environment, the operator would be responsible for the performance of up to three growers. This responsibility relies heavily on the skill and dedication of the operator. The potential for operator induced error adversely affecting crystal growth yields is therefore quite high. It is considered that all of the sequential type operations lend themselves ideally suited to microprocessor control and so eliminate operator induced error.

The program objective therefore deals primarily with reduced cost by allowing the crystal puller/operator ratio to be increased and the improvement of growth yields by the reduction of operator error.

The development of an automated growth process was undertaken using a Motorola Exorcisor microprocessor system. All necessary programming and software development was done in-house together with the wiring modifications to the puller control console to allow the interfacing of the total system to the Hamco CG2000 RC crystal grower.

The microprocessor interfaced with the CG2000 RC crystal grower is illustrated in Figure 14.

Work continued in programming and development of the software. By the end of the first quarter of 1980, the following process sequences were developed and demonstrated:

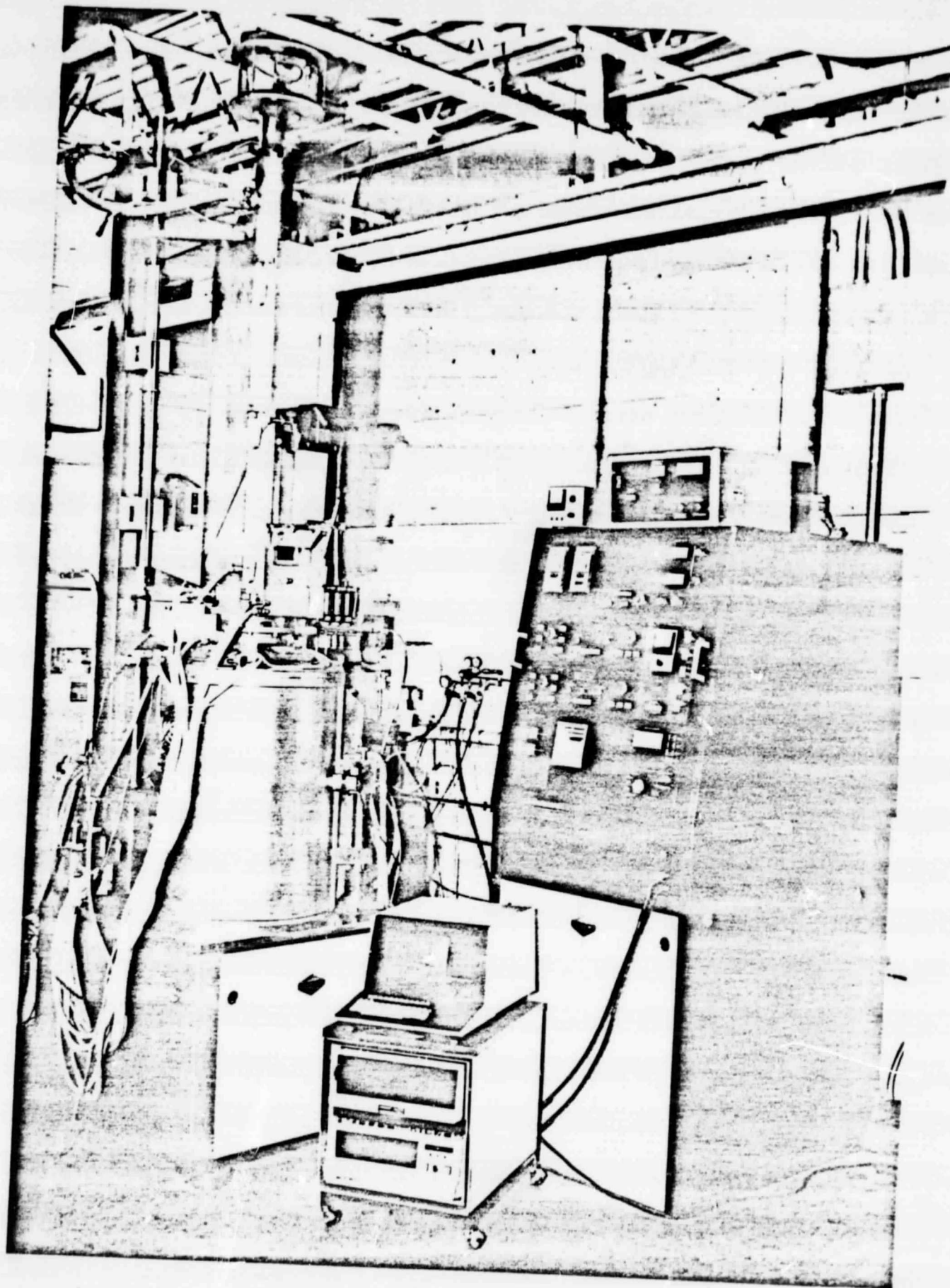
- a) initial melt-down
- b) stabilization of the melt temperature
- c) all motor control functions
- d) manual control of the crystal growth process through the microprocessor
- e) bench testing of the automatic and diameter growth cycles through the microprocessor were partially complete.

A detailed program plan outline was prepared at this time and is illustrated in Figures 15a through 15g.

During April, 1980, a technical direction memorandum was issued by JPL giving the development of a microprocessor control of the crystal growth process as the number one priority. The crystal grower utilized in this project was made totally available for this section of the project during the months of April, May and part of June.

A demonstration of microprocessor controlled growth was scheduled initially for the week ending May 2, 1980. A fault developed in the A/D printed circuit board which, at first, was thought to require replacement. Prolonged bench testing failed to reproduce the fault. Interrupts to the memory system also caused problems. The memory was diagnostically tested for bad cells and 35V (peak-to-peak) square wave pulses were observed on the A.C. line. A power line conditioner was obtained and incorporated into the system. The pulses were traced back to the power supply used in the Hamco CG2000 RC crystal grower. Both of these problems necessitated re-scheduling of the demonstration to May 19, 1980.

Prior to the above discussed problems, a series of crystal growth runs were made so that microprocessor interfacing and various control systems could be checked, calibrated and adjusted as necessary. All of these evaluation runs were performed using 12 inch diameter quartz crucibles and ancillary graphite piece parts to conserve cost. The first run (No. 11) was made April 23. The neck and crown were grown utilizing control through the MPU. Calibration values were established for all



MPU INTERFACED WITH CG2000 RC CRYSTAL GROWER

Figure 14

PROGRAM OUTLINE

1. Control I

A. Goals

1. Prompt operator for proper sequence of operation.
2. Cause "crucible rotation" to be under control.
 - A. Motor control routine
3. Cause "crucible lift" to position crucible.
4. Perform "bakeout" by operator determined parameters.
 - A. Temperature level (entered)
 - B. Soak time (entered)
 - C. Crucible position (entered)
5. Perform "meltdown" by operator determined parameters.
 - A. Temperature level (entered) meltdown
 - B. Soak time (entered) meltdown
 - C. Crucible position (entered) meltdown
 - D. Temperature level (entered) stabilize
 - E. Soak time (entered) stabilize
 - F. Crucible position (entered) stabilize
6. Monitor alarm sensors and shutdown if major.
 - A. Water flows (major)
 - B. Water temperatures (minor)
 - C. Pressures (minor/rapid rise major)
 - D. Positioning (minor)

B. Minimum Acceptance

1. Prompting
2. Crucible motions
3. Auto; bakeout, meltdown and stabilization

Figure 15a

II. Control II

A. Goals

1. Allow control of all motors (speed only).
 - A. Crucible lift and rotation (no jog)
 - B. Seed lift and rotation (no jog)
2. Allow for temperature variations.
3. Routine exit.
 - A. Abort by operator
 - B. Abort due to major alarm
 - C. Exit to auto-diameter control

B. Minimum Acceptance

1. All motors under control
2. Temperature variations possible
3. Operator to be able to perform manual growth
 - A. Dip seed
 - B. Grow neck
 - C. Grow crown
 - D. Shoulder crystal
 - E. Abort by operator or exit to auto (this stage still aborts)

Figure 15b

III. Control III

A. Goals

1. Control seed lift via diameter input.
 - A. Operator can change diameter required.
 - B. Reticon (or photocells if Reticon unacceptable) diameter input.
(Reticon installed and tested).
2. Lockout operator attempts to change rotational speeds.
Operator may abort or exit to auto or manual.
3. Crucible lift a function of SL, cal. xtal weight, and crucible size.
4. Aborts due to major alarm.

B. Minimum acceptance

1. Control seed lift by diameter input.
2. Slave crucible lift to SL, xtal weight, and crucible size.
3. Lockout unacceptable operator commands.
 - A. Rotational speed changes. (Abort or full auto or man. allowed).

Figure 15c

IV. Control IV

A. Goals

1. Increase and decrease temperature set point as a function of the average deviation of the seed lift from the seed lift set point.
2. Operator allowed to:
 - A. Abort
 - B. Exit manual
 - C. Exit auto diameter
 - D. Change SL or diameter set points
3. Abort due to major alarm.

B. Minimum acceptance

1. Temperature set point a function of average seed lift deviation from its set point.
2. Operator may abort on exit to Controls II or III.
3. Operator may change seed lift or diameter set points.

Figure 15d

V. CG2000 RC Usage

A. Simulated Runs

1. Requires all machine functions except for temperature and diameter sensing.
- 2. Temperature changes performed by monitoring the appropriate D/A output.
(Compare voltage to an actual value for the same set point reading).
3. Diameter testing by various forms of light sources.
4. Test actual motor speeds versus required and displayed actual.

B. Actual Runs

1. Requires:
 - A. Bakeout - from cold machine
 - B. Meltdown - from finish of bakeout
 - C. Various stages of actual crystal growth, i.e. neck only or neck and crown, etc.
2. Grower should not be in use for more than one shift in most cases.

Figure 15e

VI. Data Storage and Process Development

A. Goals

1. To store run data at fixed time intervals (und. as yet).
 - A. All motor settings
 - B. All motor tach readings
 - C. Diameter setting
 - D. Temperature set point
 - E. Actual diameter (req. Reticon or similar)
2. To store run data when operator causes a change to occur, i.e. enters new set point.
3. Produce a hard copy of all run data from the floppy disk.
 - A. Suitable format to be used for ease in analyses of data

B. Minimum Acceptance

1. Run data storage on floppy disk.

Figure 15f

VII. Programmed Growth

A. Goals

1. Allow operator to enter run data points.
 - A. Particular data points, i.e. SL, CR, etc.
 - B. Parameters for use of data points, i.e. time into run or xtal weight or both, etc.
2. Allow for permanent storage of data points.
3. Retrieval of stored for use in successive runs.
 - A. Eliminates need for operator entry at start of each run.
4. Allow operator to edit and change growth program.
5. All entry and edit features to be in plain English and engineering units so as to require no programming knowledge on the operator's part.

B. Minimum Acceptance

1. All goals listed previously.
2. Option to postpone until later date due to process development problems.
3. If postponed, then some form of programmed tailing operation of crystal to be developed to JPL contract requirements.

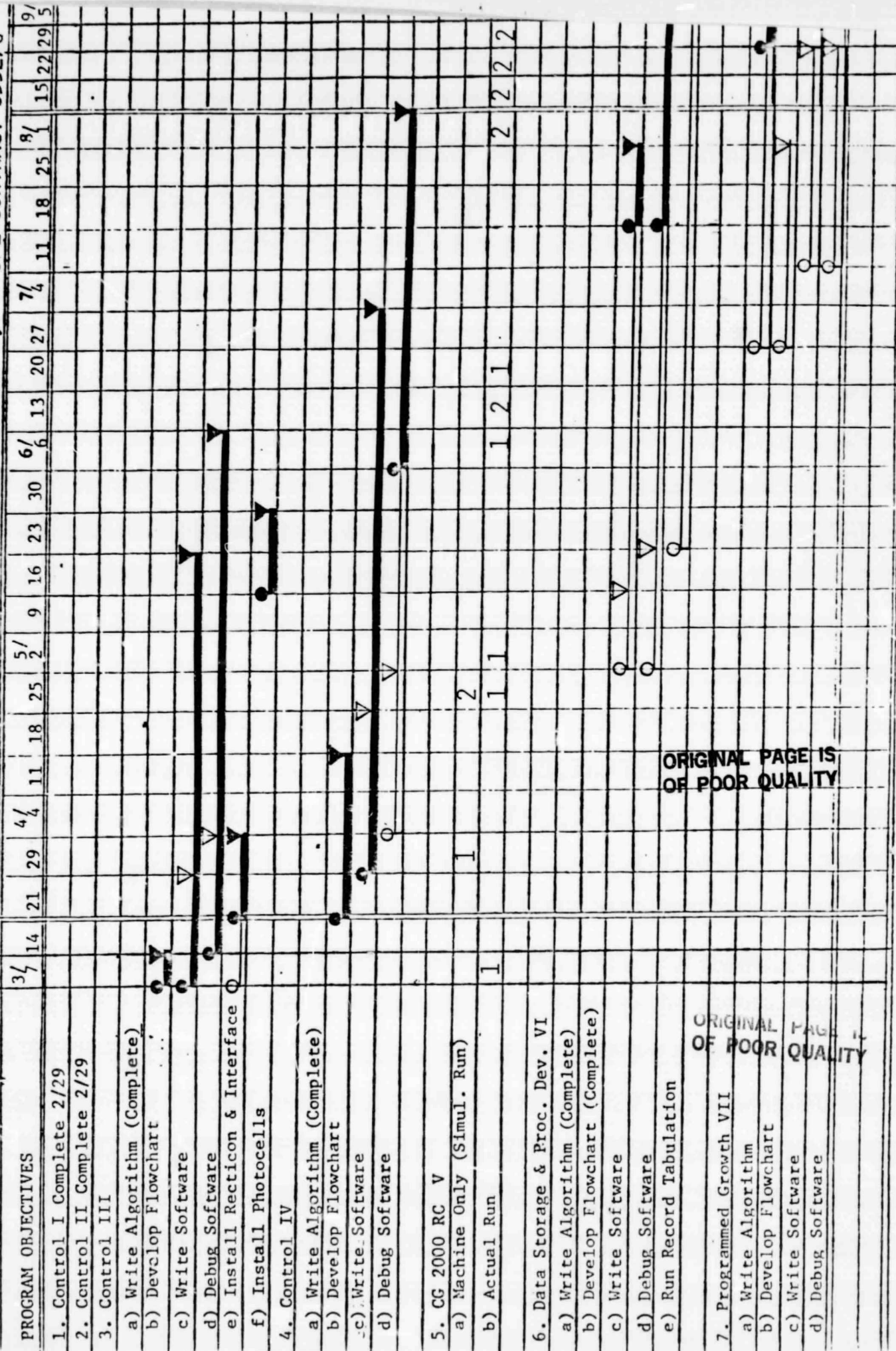
Figure 15g

MICROPROCESSOR CONTROL PROGRAM PLAN

LSA PROJECT

LARGE AREA SILICON SHEET

JPL CONTRACT 955270



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REPORT DATE: SEPT. 24, 1980

Figure 16

Kayex Corporation, Rochester, NY

motors. Software adjustments were made such that the motor control sequence would be displayed first.

Dry runs were made on April 25 and 29 to check out previous corrections and to implement the seed travel and crucible travel interface.

Run #12 was made April 30 when the heater was initially powered manually and then actual melt down and melt stabilization was achieved through the MPU.

Run #13 was made May 6 to check out and comprehend all the corrections that were made were operable.

Run #14 was made May 8 when the MPU was interfaced parallel with the crystal grower. The seed and crown were grown to the shoulder by adjusting the puller control sequencing through the microprocessor. Adjustments were made to alter the display sequencing from 3 seconds to 1 second.

Two further crystal growth runs, i.e. Nos. 15 and 16, were made in an attempt to integrate the Reticon camera system into the second loop (growth control) program. Software malfunction was traced to the Reticon system giving an imaging problem causing false diameter readouts. This created severe diameter control problems resulting in a decision being made to abandon the Reticon control system and to utilize the standard Hamco CG2000 RC crystal grower photocell diameter sensing technique. This changeover was completed June 6, 1980.

At this time, a critical review of the program was undertaken. It was felt that the MPU program was utilizing the CG2000 puller full time and so preventing development work associated with the accelerated growth program from continuing. A decision was made to provide an additional crystal grower to the project at Kayex expense for microprocessor development. Microprocessor interface wiring to this grower was commenced and was completed during July, 1980.

A revised microprocessor control program plan was generated and is illustrated in Figure 16. The illustration shows the program plan at contract completion.

During the final quarter of the program (July - September, 1980), a concentrated effort was made to finalize the microprocessor sequencing to control total crystal straight growth parameters.

Tuning of the second loop (growth control in the MPU) and the automatic diameter control in the 3 mode controller was done alongside a total of eight crystal growth runs.

The system was integrated on the sixth crystal growth run. A 4" diameter crystal was grown with a slight amount of diameter variation. Further tuning was performed and a successful 4" diameter crystal was grown on crystal run number eight.

The microprocessor system was reverted back to the contract crystal puller for demonstration of 6 inch diameter crystal growth as defined in the contract

objectives.

Two further crystal growth runs were performed on the contract crystal puller to demonstrate microprocessor control.

Crystal growth run number 31 was performed September 8, 1980. Manual control of the shoulder sequencing through the microprocessor followed by microprocessor control of the straight growth was demonstrated for a crystal weight of 12 kg at 150 mm diameter. Growth was terminated at 12 kg due to a viewport window cracking and creating a substantial air leak.

A further crystal growth run, number 32, was commenced September 10, 1980. The goal of this crystal growth run was to produce 150 kg of silicon crystal from one quartz crucible by recharging and utilizing microprocessor control of the growth process.

A total of three crystals were grown utilizing previously described recharging techniques. A total of 72 kg of crystal was grown and results are included in Table 1. The run was terminated after the growth of the third crystal due to the failure of the crucible. Investigation of the crucible wall thickness showed that the actual wall thickness was considerably below tolerance. The failed crucible was significantly below tolerance at the corner radius area and is considered to be the total reason for the failure.

During Run number 32, the first crystal was grown using the microprocessor to control the shoulder and straight growth. The second loop (growth controller) was not entered; consequently, no temperature additions were made. This resulted in a freeze-out occurring after 21 kg of crystal growth.

The second crystal was grown as standard (without microprocessor control) to enable necessary adjustments to be made to the second loop. A crystal totalling 26.2 kg was grown.

The third crystal grown was grown utilizing total microprocessor control of the straight growth portion of the run. Taper and shouldering of the crystal was performed using manual input through the microprocessor. The crystal weight produced was 24.8 kg. Total weight of crystal grown on Run #32 was 72 kg.

The status of the microprocessor automation program at the end of the project is as follows:

- 1) Microprocessor control of the crystal growing "straight growth" process has been demonstrated for both 4" and 6" diameter crystal.
- 2) Manual control of the seeding, neck growth, taper and shouldering processes through the microprocessor has been demonstrated.
- 3) Microprocessor control of the melting and melt stabilization process using

CRYSTAL GROWTH RUN #32

Crystal ID# Run-Xtal #	Crystal Length (in)	Crystal Weight (kg)	Pt. of Dislocation (in)	%OD	Inches of Single Xtal (in)	% of Single Xtal	St. Growth (hrs)	Growth Rate St. Growth	Total-Single Xtal % of Run
32-1	22-1/2 + 0	21.0	11	48.9	13	57.8	8.7	2.59"/hr	32.2
32-2	27 + 0	26.2	1-1/2	5.6	6	22.2	9.9	6.58 cm/hr 2.73"/hr	
32-3	28-1/4 + 0	24.8	1	3.5	6	21.2	9.9	6.93 cm/hr 2.85"/hr	
Total	77.75	72			25		28.5	7.24 cm/hr 2.73"/hr	

TABLE 1

operator prompting has been demonstrated.

4. Manual control of the "taper out" process through the microprocessor has been demonstrated.

It is considered that the feasibility of microprocessor control has been accomplished. However, the achievement of effective cost reduction and yield improvement by the utilization of total automation of the growth process requires additional development.

2.3 Accelerated Growth Rate Program

The accelerated growth rate concept was incorporated into the overall program as a potential for increasing the throughput and lowering the cost.

It was felt that, by employing a suitable heat sink arrangement, the energy released by the heat of fusion accompanying the transformation of silicon from the liquid to the crystalline state could be removed. Prior random experimentation suggested that increased growth rates of >10 cm per hour could be achieved. If this was the case, it was felt that an approximate 18% cost reduction could be achieved by increasing the average growth rate from 10 cm per hour to 15 cm per hour. This is illustrated in Figure 17. It is further illustrated that further increases in crystal growth rate become less influential in reducing the cost per kilogram because a number of other costs are unchanged. Using SAMICS/IPEG guidelines, the reduction in cost by increasing crystal growth rate is due to decreased labor cost and increased throughput.

Initially, it was considered practical to utilize the water cooled copper RF coil used for the polycrystalline rod melting as the heat sink arrangement to accelerate crystal growth.

The RF coil was designed and fabricated to be adjustable through the vertical plane. After completion of the poly rod melting sequence, the work coil was isolated from the RF generator and adjusted as necessary through the vertical plane to operate as a heat sink.

Utilization of the water cooled RF coil as a heat sink caused problems relating to heavy silicon oxide deposition on the coil. The oxide continually built up on the coil and then fell into the melt causing immediate crystal structure loss.

A summary of the results of crystal growth run number 4, which utilized the water cooled RF coil concept as a heat sink arrangement, is included in Table 2. together with a photograph illustrating the technique in Figure 18. The photograph illustrates crystal number 1 during growth through the heat sink.

The crystal diameter grown during run number 4 was 5" (12.7 cm). The crystal growth rates achieved were 4.12"/hr (10.46 cm) and 4.6"/hr (11.68 cm) respectively

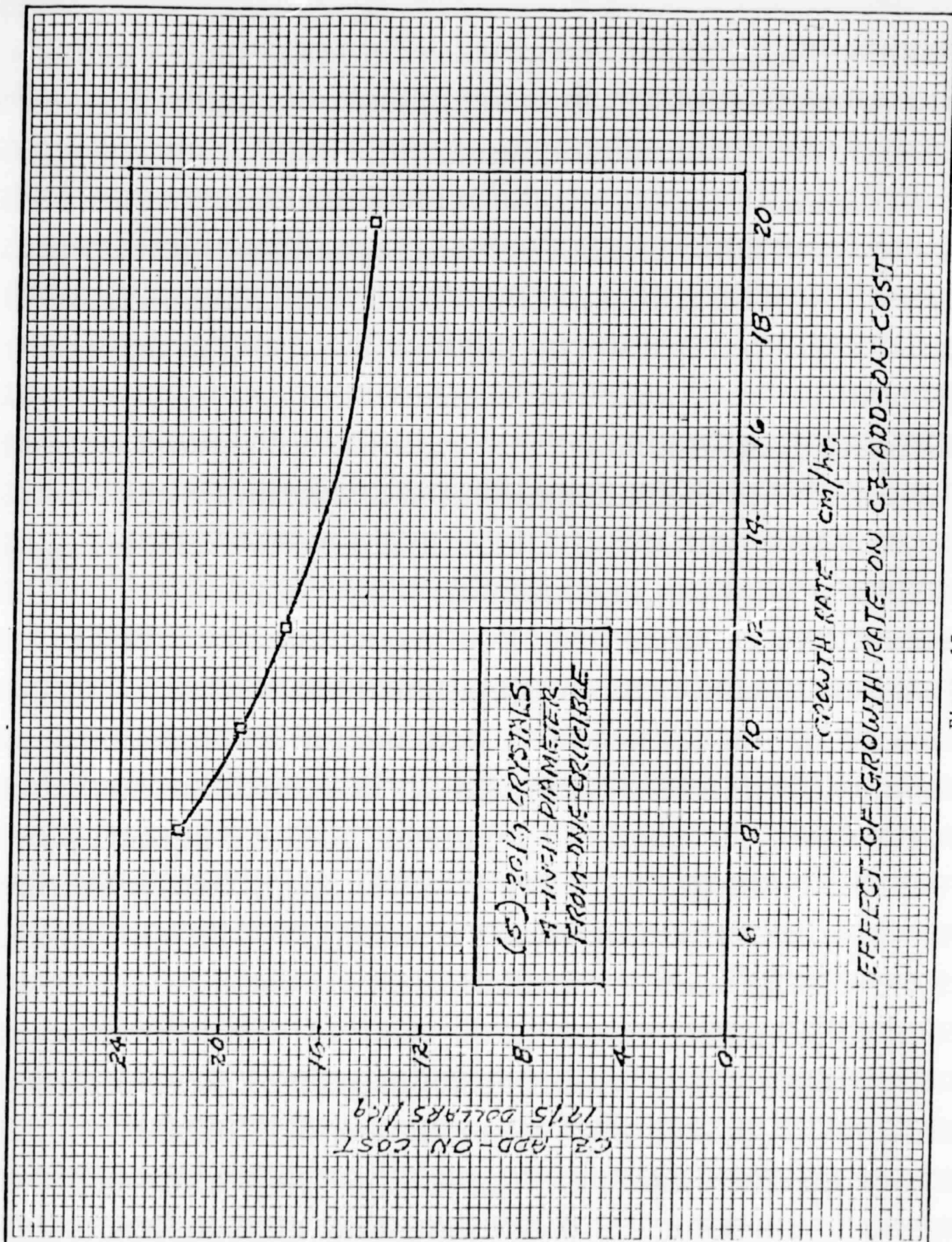


Figure 17

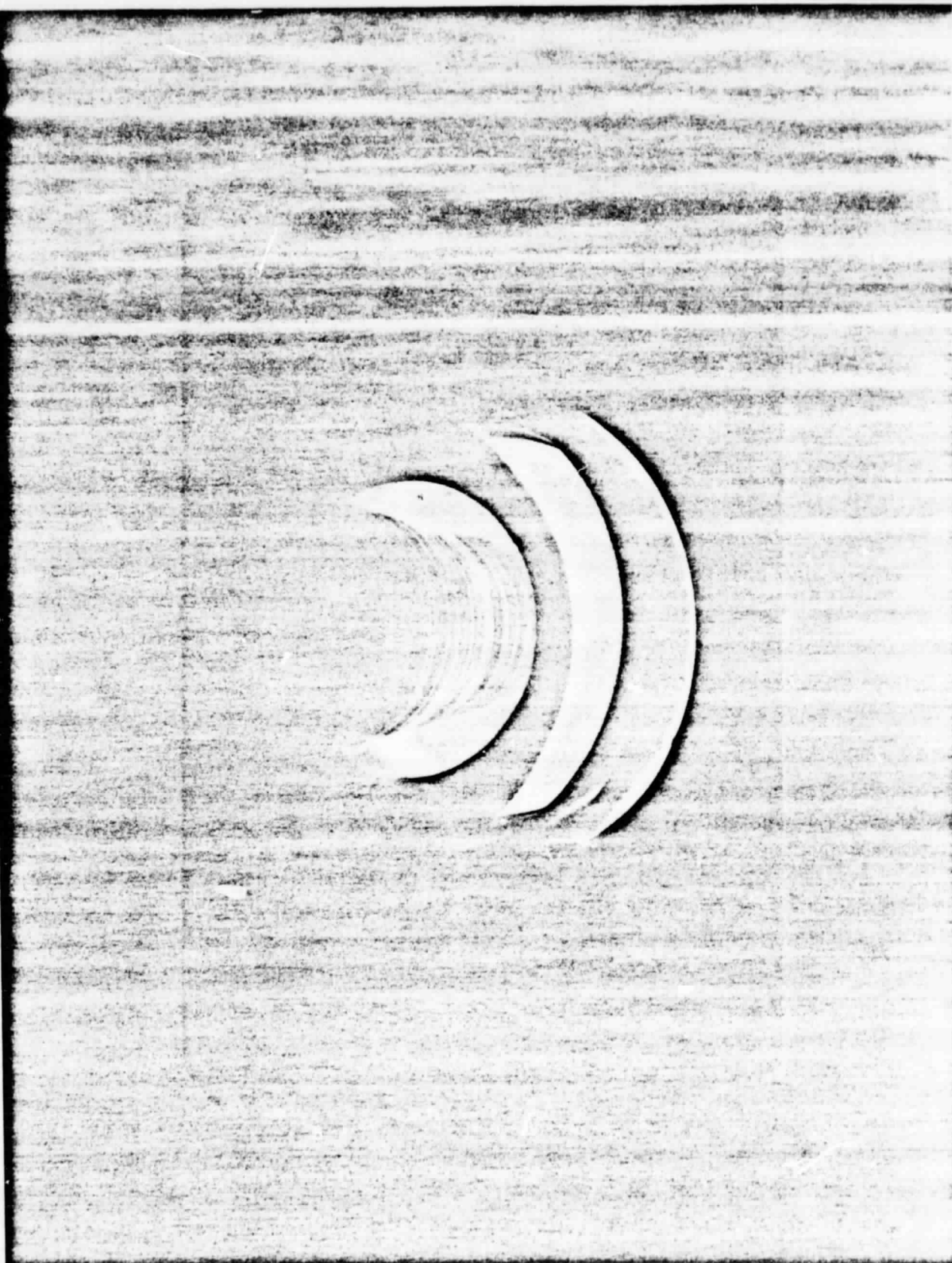
SUMMARY OF RUN #4

Run was completed using 12" piece parts.
 Crystal diameter pulled was 12.7 cm.
 Initial charge weight = 17.6 kg.
 Poly rod recharge weight = 12.8 kg.

CRYSTAL #	CRYSTAL LENGTH	CRYSTAL WEIGHT	PT. OF DISLOCAT.	% O.D.	INCHES OF SINGLE XTAL	% OF SINGLE XTAL	ST. GROWTH (HRS)	GROWTH RATE
1	17.5	12.6 kg	12"	68.6%	13.5"	77.1%	4.25	4.12"/hr = 10.46 cm/hr
2	11.5	8.1 kg	2"	17.4%	4.5"	39%	2.5	4.6"/hr = 11.68 cm/hr

Total poly rod recharge time (from completion of taper to subsequent seed dip):
 Total recharge time to seed dip = 3 hrs.
 Total recharge time to straight growth = 4 hrs. 50 min.

TABLE 2



Crystal Growth Using Water-Cooled RF Coil as a Heat Sink
Figure 18

for crystals Nos. 1 and 2.

Due to the oxide flaking problem, it was apparent that the design and development of an alternative heat sink system was necessary to prevent oxide build-up and flaking.

A conical shaped molybdenum heat shield was fabricated for use with a 14-inch diameter crucible set-up. The system was installed initially in a different Hamco crystal puller from the contract puller so that the microprocessor development could continue uninterrupted. A crystal growth run (noted as special radiation shield development run) was undertaken on May 20, 1980 utilizing the new 14-inch set-up. An initial charge of 26.2 kg was cold charged into an Amersil crucible. Problems were encountered in establishing the optimum crucible start position in relation to heat shield distance from the melt level. An initial crystal, 5-1/2 inches in length, was pulled from the melt to establish parameters. Once these parameters were set, crystal No. 1 was removed from the grower, and growth commenced on crystal No. 2. Crystal diameter pulled was 5.3 inches. A total of 15.6 kg of material was grown in 7.66 hours (from seed dip to taper out of the crystal) representing a total throughput of 2.03 kg per hour.

The initial heat shield design required some modification, primarily to the positioning method, to insure optimization of the thermal conditions. One immediate advantage of using this form of heat shield was that it readjusted the argon flow pattern such that: a) the crystals produced were totally free of any oxide formation down their length, b) significant improvement was made to the amount of oxide formation on the crucible wall freeboard and rim. It was thought that this could have a significant impact on the ability to grow multiple dislocation-free crystals.

The contract CG2000 RC crystal grower was converted to 14-inch piece parts and a crystal growth run (Run No. 19) performed on May 29, 1980. The purpose of this run was to establish parameters for 150 mm diameter growth on this puller from 14-inch diameter piece parts. From a charge of 30.14 kg, a total of 24.5 kg was pulled in a total overall time of 11.1 hours. However, a diameter control problem existed throughout the run. The diameter progressively tapered from 5.8 inches to 6.4 inches through its length. This explains the higher throughput rate of 2.21 kg per hour. Approximately 10.4 kg of crystal was zero dislocation.

A new molybdenum heat shield was fabricated and installed in the puller. The heat shield was tapered at an angle of approximately 21 degrees and had a lower diameter of 7-1/2 inches. This would allow 6-inch diameter crystals to be pulled through. The overall length allowed for a starting melt level of 2 inches below the rim of the heater for a 30 kg charge in a 14-inch diameter crucible. Also, a design

using three locating and positioning feet was built into the system.

Four crystal growth runs (Nos. 20, 21, 22 and 23) were made during the month of June. Nos. 20 and 21 were single runs and Nos. 22 and 23 were multiple runs. All runs, with the exception of Run No. 21, produced high zero-dislocation yields at relatively improved throughput rates. An example of the improved cleanliness of the crucible wall obtained is illustrated in Figure 19. The section on the left illustrates oxide build-up after normal growth without the heat sink. The section on the right illustrates the relative cleanliness of the crucible wall after 37.5 hours of multiple growth (Run No. 23).

An illustration of the heat shield is shown in Figure 20.

Details of crystal growth runs are included in Tables 3 and 4. To date, cold filling of 30 kg of silicon into the crucible has not been attempted with the heat sink arrangement in position, but no problems are anticipated. Approximately 20 kg was cold filled and melted and the remaining 10 kg was hot filled utilizing developed hopper recharging techniques to get the 30 kg melt. The crystals grown during multiple crystal growth run No. 23 are illustrated in Figure 21.

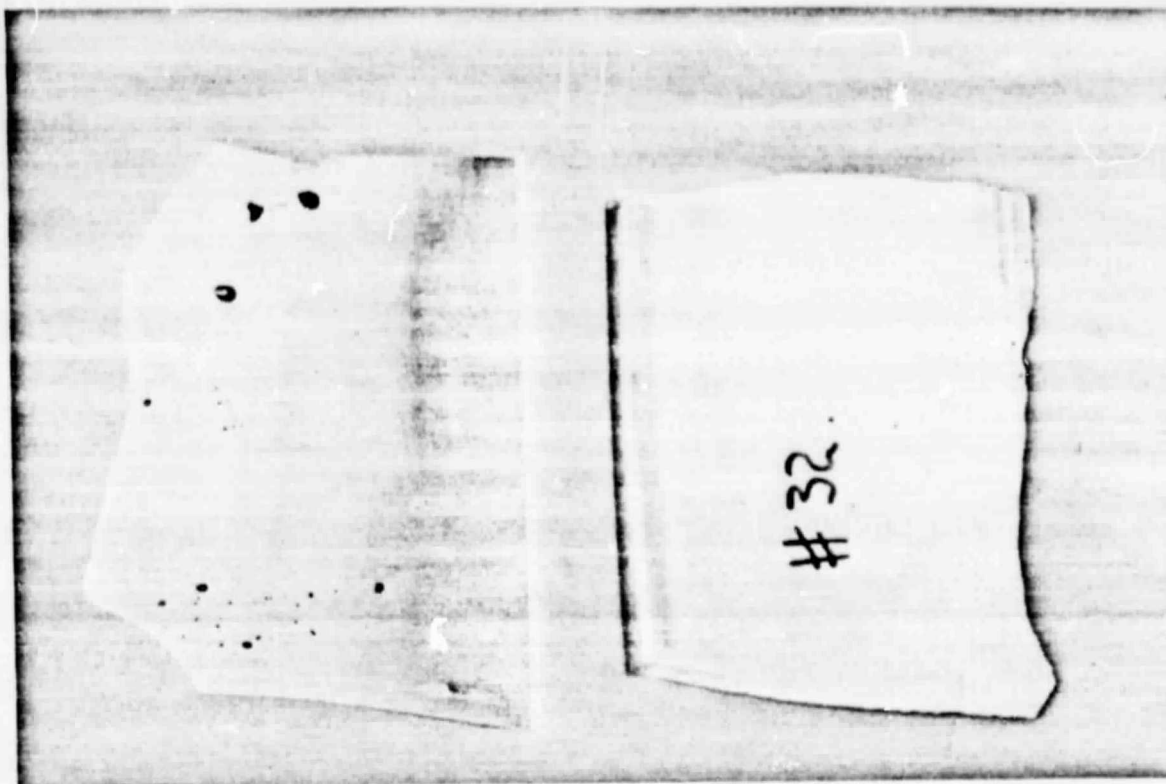
During the accelerated growth program process development using the radiation shield, some problems occurred relating to reduced visibility of the crucible wall during crystal growth. As the radiation shield position was fixed in the grower and the crucible travels upwards during the growth cycle, the view of the quartz crucible walls becomes progressively reduced during growth. A schematic of the system readily illustrating the problem is shown in Figure 22.

To overcome this problem, a series of slots were placed in the radiation shield. Visibility was slightly improved, but the argon flow pattern was altered considerably. This contributed to an increase in the amount of oxide that was deposited on the rim of the crucible. This deposit had the potential of falling into the melt, which could cause a structure loss problem.

A new shield was fabricated with just one slot placed in it to allow the photocell diameter control system to sight the meniscus.

Several further growth runs were made including a 150 kg recharge run (crystal growth run number 30) up to the end of the contract. Details of growth run numbers 27, 30 and 32 are shown in Tables 5, 6, and 7.

The highest straight growth rate achieved was 3.88 inches per hour (growth run 27, crystal number 3) for 6 inch diameter crystal. This represents a straight growth throughput rate of 4.07 kg per hour. A comparison of six inch diameter growth rates achieved using standard growth conditions and radiation shield growth conditions is shown in Table 8. It can be seen that the average total growth rate achieved in crystal growth run number 72 was 2.60 inches per hour, but in crystal growth run

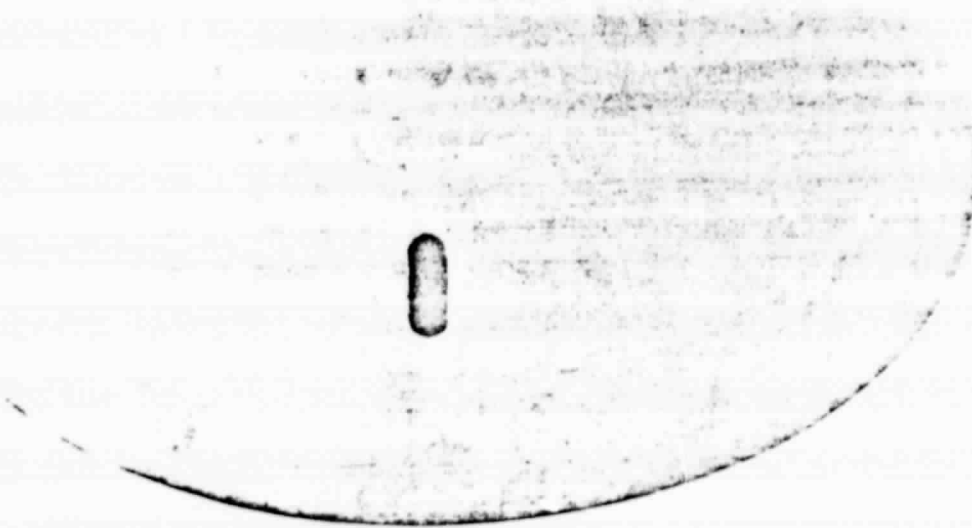
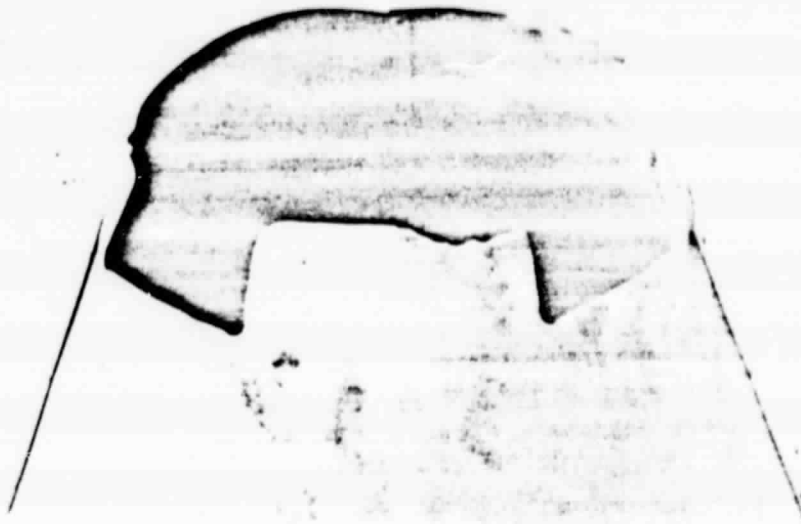


Crucible 1

Crucible 2
(37.5 hrs continuous operation)

Comparison of Oxide Formation on Crucible Sections and Rims

Figure 19



Molybdenum Heat Sink
Figure 20
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RUN NO. 20

Crystal #	Crystal Length (in)	Crystal Wt (kg)	Pt. of Dislocation (in)	% OD	Pt. of twin or Poly (in)	% Good
1	20"	19.45	-	100%	-	100%

Cold charge = 26.9 kg

Pulled yield = 72.3

Time start = 9:30 AM

Power off = 10:00 PM

Total run time = 12-1/2 hrs

Overall throughput = 1.56/hr

Straight growth start 3:00 - 10:00 = 7 hours

Straight growth throughput = 2.78 kg/hr

RUN NO. 21

Crystal #	Crystal Length (in)	Crystal Wt (kg)	Pt. of Dislocation (in)	% OD	Pt. of twin or Poly (in)	% Good
1	27.5"	27.7	7"	25.4%	9" Nucleation on crucible wall occurred at 5"/hr growth rate. Resulted in loss of OD.	32.7%

Cold charge = 18 kg

Hot fill = 12 kg

Total = 30 kg

Time start = 8:35 AM

Time finished = 1:00 AM

Total run time - 16.65 hrs

Pulled yield = 92.3%

Overall throughput = 1.66 kg/hr

Straight growth start 5:00 PM - 1:00 AM = 8 hours

*Difficulty establishing thermal parameters from 11:00 AM until 3:00 PM

Straight growth throughput = 3.46 kg/hr

RUN NO. 22

Crystal #	Crystal Length (in)	Crystal Wt (kg)	Pt. of Dislocation (in)	% OD	Pt. of twin or Poly (in)	% Good
1	24-1/2"	22.8	None	100%	-	100%
2	16-1/4"	14.2	None	100%	-	100%
Totals	40-1/2"	37.0		100%		100%

RUN NO. 22 (cont'd)

Time start = 8:33 AM 6/19/80 Time finished = 10:20 AM 6/20/80
 Total run time = 25-3/4 hours Total throughput = 1.44 kg/hr

Crystal #1 (90% of crystal grown manually)
 Straight growth 4:45 PM - 12:05 AM = 7.3 hours
 Straight growth throughput = 3.12 kg/hr

Crystal #2
 Straight growth 5:30 AM - 10:20 AM = 4.8 hours
 Straight growth throughput = 2.94 kg/hr; average pull speed = 3.36"/hr or 8.53 cm/hr

RUN NO. 23

Crystal #	Crystal Length (in)	Crystal Wt (kg)	Pt. of Dislocation (in)	% OD	Pt. of twin or Poly (in)	% Good
1	24" + 2" taper	22.2	None	100%	-	100%
2	18.25" + 4" taper	18.7	16"	87.7%	18"	98.6%
3	21.25" (no taper)	18.6	15.25"	71.8%	17"	80.0%
Total	63.5" +	59.5 (4.5 kg est. for tapers = 55 kg)	55.25"	86.5%	59"	92.9%

Total charged = 65.4 kg Wt pulled = 59.5 kg
 Pull yield = 91%
 Total run time 8:35 AM - 10:10 PM = 37.4 hours
 Total throughput = 1.59 kg/hr

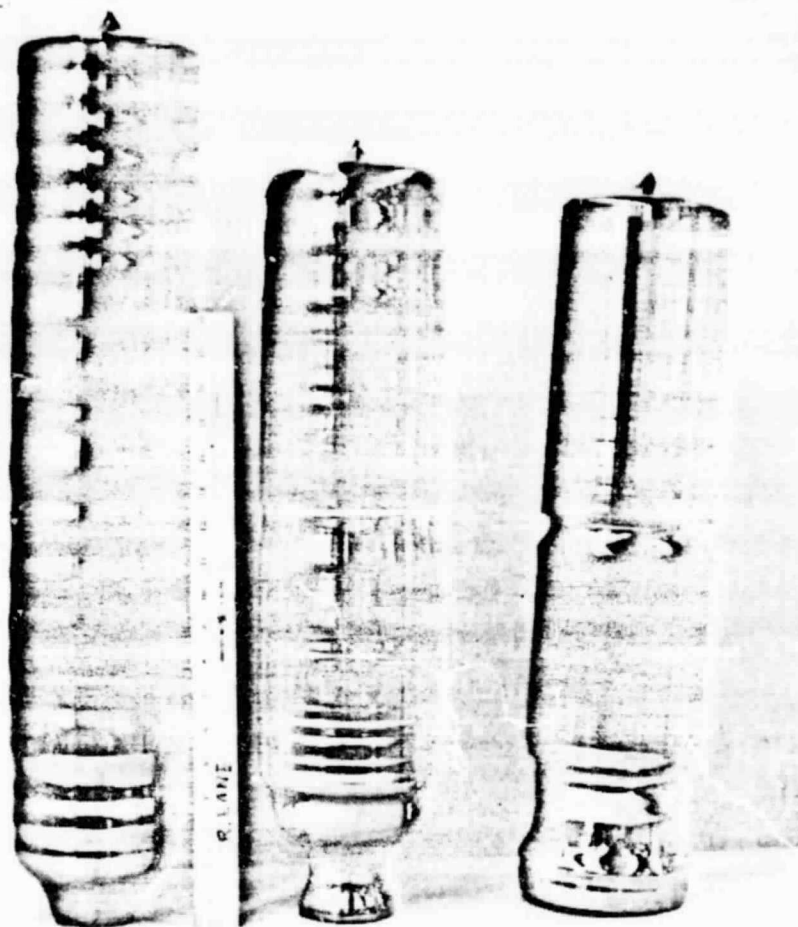
Crystal #1
 21 kg in 7.8 hours
 Straight growth throughput = 2.69 kg/hr = 3.06"/hr or 7.7 cm/hr

Crystal #2
 16.7 kg in 5 hours
 Straight growth throughput = 3.34 kg/hr = 3.65"/hr or 9.27 cm/hr

Crystal #3
 14.8 kg in 5.5 hours
 Straight growth throughput = 2.69 kg/hr = 3.09"/hr or 7.85 cm/hr

Straight growth calculations for Crystals 1 and 2 exclude (estimated) taper out lengths and weights. No. 3 was not tapered out of the melt.

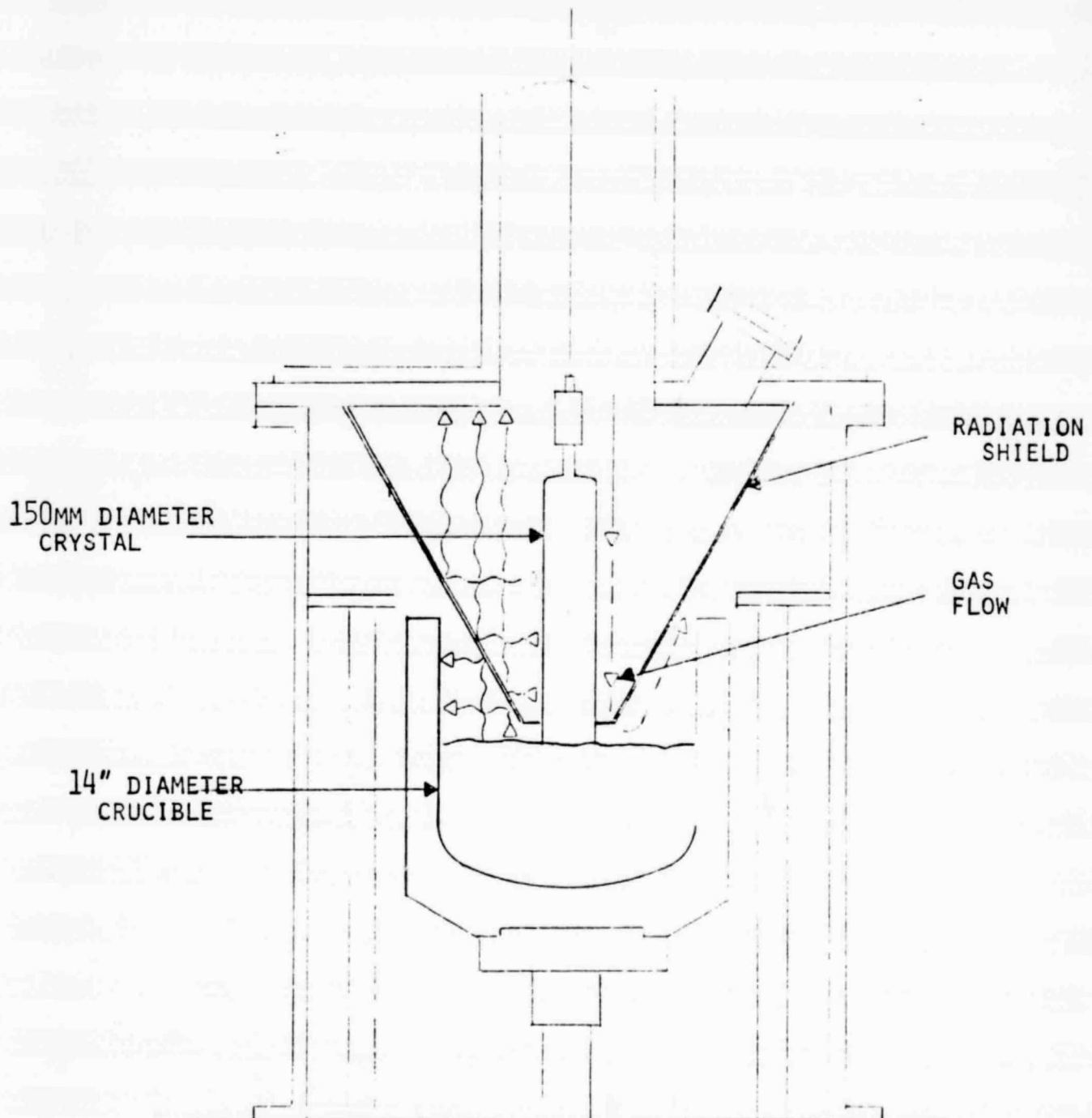
TABLE 4



Crystals Grown - Run #23

Figure 21

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LARGE AREA SILICON SHEET
KAYEX CORPORATION JPL CONTRACT NO. 955270
CZ FURNACE RADIATION SHIELD



REPORT DATE: SEPT. 24, 1980
START DATE; MARCH 12, 1979

Figure 22

Crystal ID# Run-Xtal #	Crystal Length (in)	Crystal Weight (kg)	Pt. of Dislocation (in)	% OD	Inches of Single Xtal (in)	% of Single Xtal	Straight Growth (hrs)	Growth Rate St. Growth	Total-Single Xtal % of Run
27-1	22" + 2-1/2" Taper	22.8	None	100	22	100	7.0	3.15"/hr 8.00 cm/hr	61.9
27-2	22" + 3/4" Taper	21.8	11	50	12	54.5	7.4	2.97"/hr 7.54 cm/hr	
27-3	15-1/2"	15.4	2-1/2	16.1	3-1/2	22.6	4.0	3.88"/hr 9.86 cm/hr	
27-4	5	5.2	Crown	0	3	60	1.6	3.13"/hr 7.95 cm/hr	
TOTAL	64.5	65.2			40.5		20.0		

TABLE 5

Crystal ID# Run-Xtal #	Crystal Length (in)	Crystal Weight (kg)	Pt. of Dislocation (in)	% OD	Inches of Single Xtal (in)	% of Single Xtal	Straight Growth (hrs)	Growth Rate St. Growth	Total-Single Xtal % of Run (First three crystals 77/2)
30-1	23-1/2 + 3" Taper	24.0	None	100	23-1/2 + Taper	100	6.7	3.51"/hr	(First three crystals 77/2)
30-2	22 + 2" Taper	24.1	10	45	13	59	6.3	3.49"/hr 8.68 cm/hr	
30-3	20-1/4 + 3/4" Taper	23.3	13	64	15	74	6.75	3.00"/hr 7.62 cm/hr	
30-4	26 + 0	26.1	Crown	0	3	11.5	9.7	2.68"/hr 6.81 cm/hr	45.6
30-5	24-1/4 + 1/2" Taper	24.7	2-1/2	10.3	7	28.9	8.5	2.85"/hr 7.24 cm/hr	
30-6	25-1/2 + 0	26.3	Crown	0	3	11.8	9.8	2.60"/hr 6.61 cm/hr	
TOTAL	141.5	148.5			64.5		47.75		

TABLE 6

Crystal ID# Run-Xtal #	Crystal Length (in)	Crystal Weight (kg)	Pt. of Dislocation (in)	% OD	Inches of Single Xtal (in)	% of Single Xtal	Straight Growth (hrs)	Growth Rate St. Growth	Total-Single Xtal % of Run
32-1	22-1/2 + 0	21.0	11	48.9	13	57.8	8.7	2.59"/hr 6.58 cm/hr	
32-2	27 + 0	26.2	1-1/2	5.6	6	22.2	9.9	2.73"/hr 6.93 cm/hr	32.2
32-3	28-1/4 + 0	24.8	1	3.5	6	21.2	9.9	2.85"/hr 7.24 cm/hr	
TOTAL	77.75	72			25		28.5		

First 9-1/2" under micro grown OD at 3.17"/hr

First 15-1/2" under micro at 3.0"/hr

TABLE 7

Six Inch Diameter Growth Rate Comparison
Standard Growth Conditions

Radiation Shield Growth Conditions

(1) Crystal ID Run-Xtal #	(2) Crystal Length (in)	(3) Straight Growth (hrs)	(4) Growth Rate St. Growth (in/hr)	(5) Avg 1st half growth rate (in/hr)	(6) Avg total run growth rate (in/hr)	(1) (1)	(2) (2)	(3) (3)	(4) (4)	(5) (5)	(6) (6)
70-1	22-3/4	8.25	2.76			30-1	23-1/2	6.7	3.51		
70-2	21-1/2	7.25	2.97			30-2	22	6.3	3.49		
70-3	24	8.50	2.82	<u>2.84</u>		30-3	20-1/4	6.75	3.00	<u>3.33</u>	
70-4	25-1/2	9.50	2.68			30-4	26	9.7	2.68		
70-5	23-1/4	9.90	2.35			30-5	24-1/4	8.5	2.85		
70-6	24	10.00	2.40		2.64	30-6	25-1/2	9.8	2.60		<u>2.96</u>
<hr/>											
72-1	21-1/2	7.50	2.87								
72-2	23	7.80	2.95								
72-3	22	8.70	2.53	<u>2.77</u>							
72-4	20	8.00	2.50								
72-5	24	9.20	2.61								
72-6	26-1/2	11.50	2.30		2.60						

TABLE 8

number 30, using a radiation shield, a total growth rate of 2.96 inches per hour was achieved. This represents a crystal growth increase of 0.36 inches per hour or a throughput increase of 0.378 kg per hour at 6 inch diameter.

On the number of crystal growth runs undertaken, utilizing a radiation shield as a heat sink to accelerate crystal growth, an increase in growth rate was achieved. Also, considerably cleaner melt conditions were realized, resulting in a reduced quantity of oxide build-up on the crucible walls and rim. This resulted in several crystal growth runs yielding high percentages of single crystal, i.e. runs #22 and 23. Crystal growth run number 30, which was a 150 kg recharge run, yielded a single crystal yield of 45.8% of crystal weight pulled. All the crystals pulled using a radiation shield were totally free of any oxide deposit on their surface. Use of the radiation shield redistributed the argon flow more effectively and resulted in argon flow to the viewport windows not being required. This gave a small cost reduction.

2.4 Induction Premelting of Lump/Granular Silicon

JPL programs are ongoing to produce polycrystalline silicon suitable for solar applications. It would appear that at least two of the technologies currently under development will have a potential of producing material in a granular form.

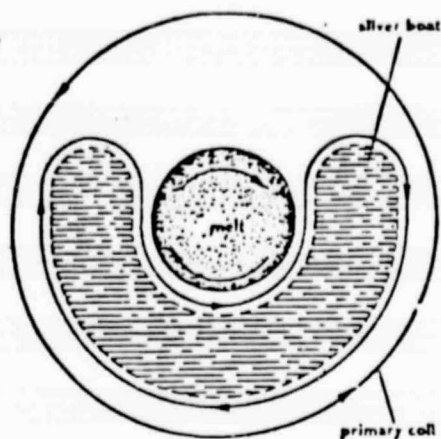
All of the development work undertaken at Kayex up to this time has dealt with crucible recharging using either polycrystalline silicon rod or chunk material. These methods have been well proven and have been shown to be contamination free, weight accurate in terms of crucible recharge and free of technological risk.⁽¹⁾

A method for recharging of the quartz crucible using polycrystalline silicon rod has been described earlier in this report together with the reasons for utilizing such a method, i.e. accelerated recharge rate to lower the add-on cost, elimination of the need to thermally cycle the crucible.

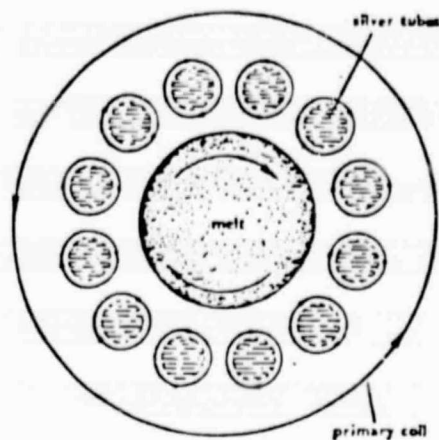
For the purpose of this program, it was necessary to identify and develop a system which would allow the crucible to be recharged using granular or graded lump silicon. This system also needed to be capable of transferring molten silicon directly into the crucible, thus eliminating the need to thermally cycle the crucible.

The technique of melting refractory materials in a cold container has been described in a paper by Sterling and Warren.⁽²⁾ They developed an induction heated silver boat arrangement for zone refining. See Figure 23. Subsequent designs of cage type cold crucibles have been developed, an example of which is the Hukin⁽³⁾ crucible, which is illustrated in Figure 24. As a result, a cold crucible silver boat arrangement was chosen for this program.

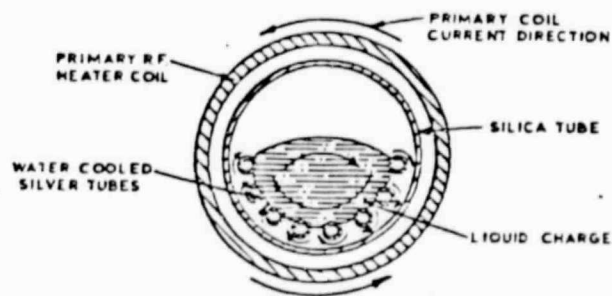
The general concept was to mechanically feed lump/granular silicon material into a



SILVER CRUCIBLE acts as an inductance coil. The primary coil induces current in the crucible, which itself induces current in the melt. Repulsion cushions the melt and hence prevents contamination



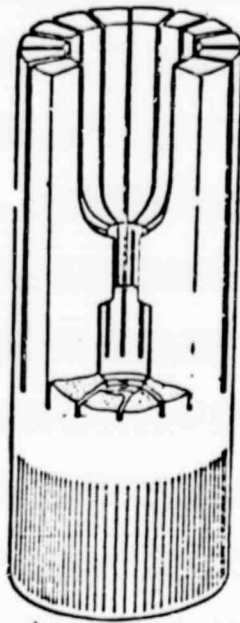
HORIZONTAL SECTION shows induction crucible for growing single crystals. Each silver tube induces current in the melt, and these currents reinforce. The tubes are thermally cold but electrically hot



Section through a horizontal silver tube boat.

SCHEMATIC ILLUSTRATIONS OF COLD CRUCIBLES

FIGURE 23



DIAGRAMMATIC SKETCH OF COLD CRUCIBLE
USED FOR CRYSTAL PULLING
(AFTER HUKIN (3))

FIGURE 24

preheater system. The material was then passed from the preheater into the RF heated silver boat assembly. The silicon was melted and levitated in the boat by the RF field. The molten silicon traversed through the boat assembly and exited directly into the quartz crucible in the Czochralski crystal puller. The weight of material transferred could be controlled by accurately pre-weighing the material in the mechanical transfer system. The rate of recharging into the Czochralski crystal grower could be controlled by varying either the melt rate or the rate of transfer of molten mass in the silver boat to the quartz crucible. This could be done by varying the angle of the silver boat. The conceptual design is illustrated in Figure 25. The RF system used in the poly rod accelerated melt program would also be used to heat the cold crucible system via a separate remote switching station and multi-turn RF coil.

The cold crucible used in this program was manufactured in oxygen free high conductivity copper and then silver plated. The crucible was initially electroplated to a thickness of 0.0003". This proved to be too thin and the assembly was replated to a thickness of 0.001". Silver was chosen because of its high electrical and thermal conductivity. It also has good reflectivity. The silver boat was surrounded by a quartz tube through which the inert gas, in this case argon, flowed. The multi-turn RF coil surrounded the cold crucible and quartz tube and was then itself surrounded by a further quartz tube.

When power is applied to the silver boat via the RF multi-turn induction coil, the silver boat acts as a single turn secondary and large currents are induced to flow. Due to the shape of the silver boat, electro-magnetic repulsion occurs between the silver boat and the silicon, resulting in levitation of the melt. The melt is therefore not in contact with the boat's surface and is free of contamination. By angling the boat slightly off the horizontal plane, the melt can be induced to flow at varying rates dependent on the angle variance.

An initial conceptual design was formulated using a silver boat arrangement having the capability of being fed with either lump or granular silicon material.

The cold crucible system was designed to be operable on a continuous melt and transfer principle during the crucible replenishment process. A storage/feed arrangement for silicon lumps would be interfaced with the cold crucible, allowing lump silicon to be continuously fed into the cold crucible. The cold crucible boat was enclosed in a quartz tube under argon pressure and was heated by a multi-turn RF work coil around the quartz tube. The RF coil would melt the silicon and, due to the induced magnetic field, the melt would be levitated and allowed to flow through the cold crucible boat. The material would then flow into a transfer

ORIGINAL COLD CRUCIBLE CONCEPTUALIZATION

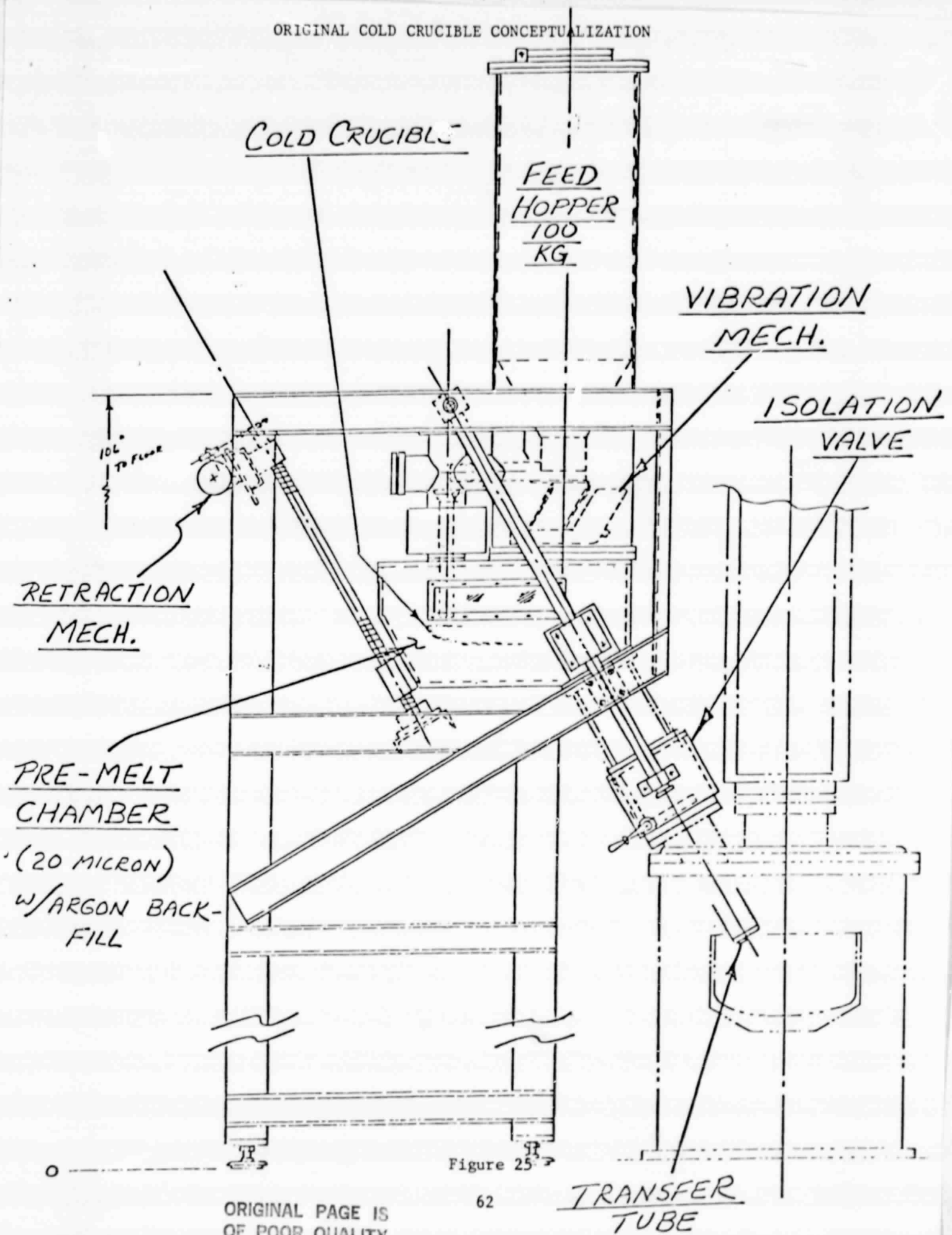


Figure 25

boat of similar construction. This boat would be heated by means of a secondary coil, which would be built within the quartz transfer tube. This total system would be fed through the tank cover into the work crucible. It would be built so as to be totally retractable, which would allow it to be adjusted after charging of the crucible such that it would not interfere with normal crystal growth.

The conceptual design was evaluated and consequently modified to allow the cold crucible to be fed into the Czochralski crystal grower via a port which was fabricated in the furnace tank wall. This redesign was necessary as it was felt that the minimum angle that could be obtained by feeding the silver boat through one of the spectator viewports in the furnace tank cover plate was too steep. The steepness of the angle would not have allowed sufficient control of the transfer of melt into the quartz crucible without causing melt perturbation and melt splashing.

A storage/feed arrangement for lump/granular silicon would be interfaced to a silicon premelter and to the cold crucible itself. Lump/granular silicon would be fed into the preheater, where the temperature of the silicon would be raised to approximately 500°C. The silicon would then pass into the silver boat and the temperature raised to melt the silicon by means of the multi-turn RF coil. The levitation principle previously described would allow the molten silicon to traverse the silver boat and so recharge the quartz crucible. The total system was designed to be retractable and so allow the system to be adjusted after completion of the recharge process so that it does not interfere with normal crystal growth. The modified system is illustrated in Figure 26.

A schematic of the silver boat/RF multi-turn coil arrangement surrounded by the appropriate quartz tubes is included in Figure 27.

The total system would be operated under an argon purge. The premelter system was assembled and operated off the crystal puller. It was necessary that the feasibility of the melt, levitate, and pour approach be demonstrated prior to the interfacing of the assembly to the crystal puller.

Initial problems developed in the high voltage remote switching RF induction heating system due to arcing between an insulation package. This was corrected, but then insufficient power output was experienced. A generator company representative was called in to rectify the fault. While making adjustments to the "tickler" or feedback coil, severe arcing occurred, causing the feedback coil to burn out. This caused just over a week's delay until a replacement was obtained.

A series of melt experiments were undertaken to evaluate and make necessary equipment adjustments. It was found necessary to fabricate some support sections for the multi-turn work coil within the quartz tube. Applying power to the RF coil while it was free standing in the quartz tube produced carbonization on the

COLD CRUCIBLE PREMELTER SYSTEM

LSA PROJECT
 LARGE AREA SILICON SHEET
 KAYEX CORPORATION
 JPL CONTRACT #955270

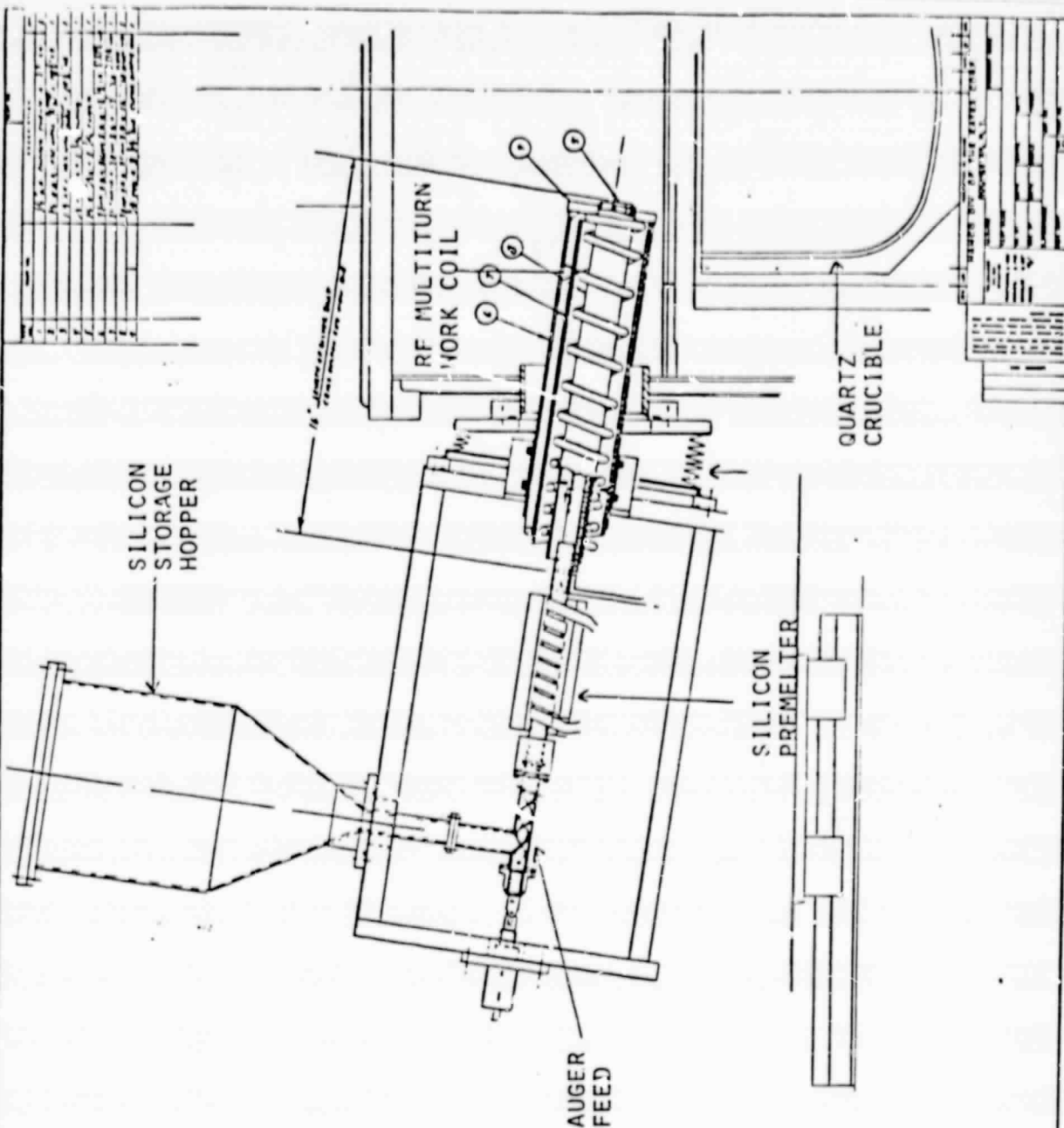
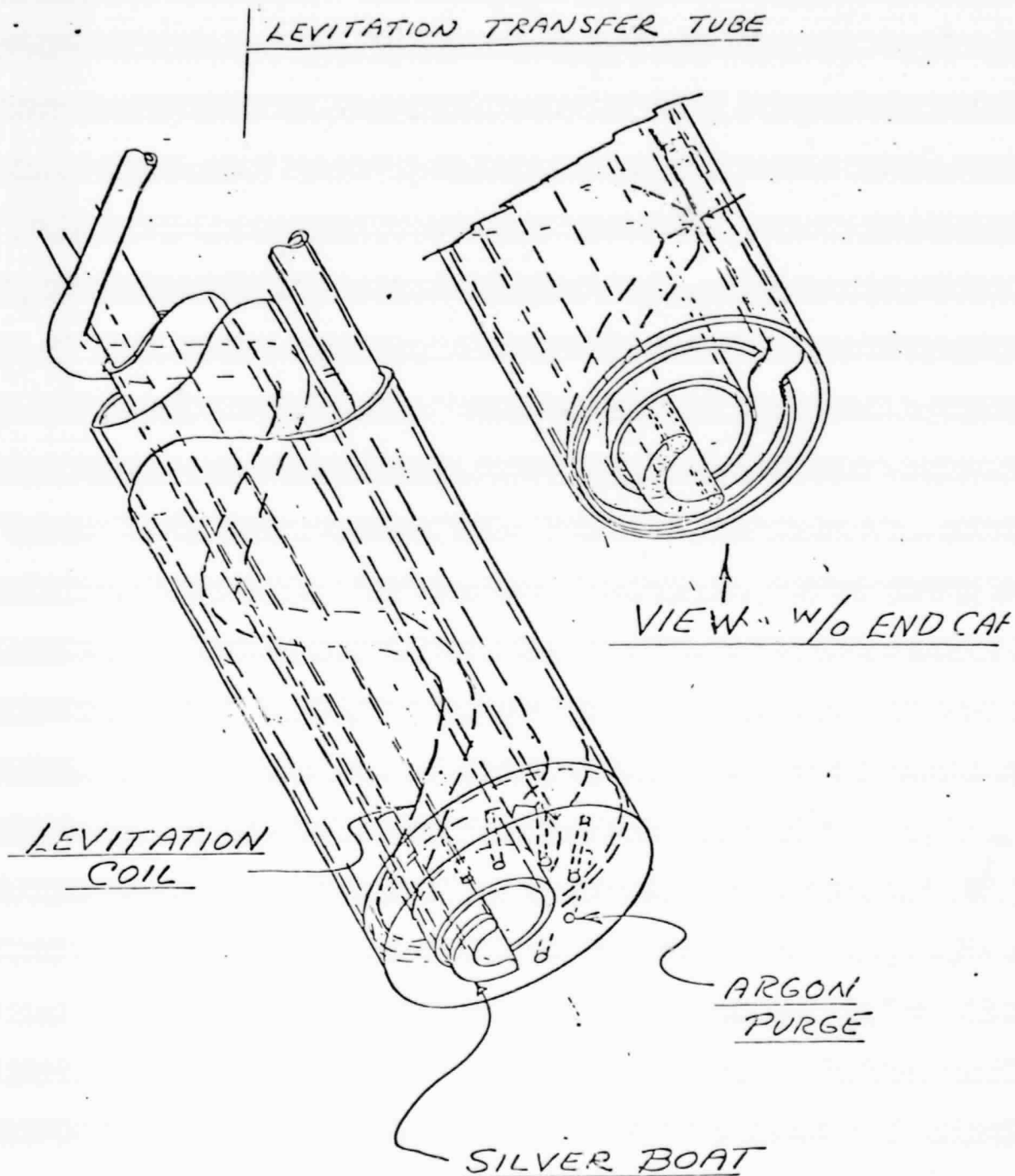
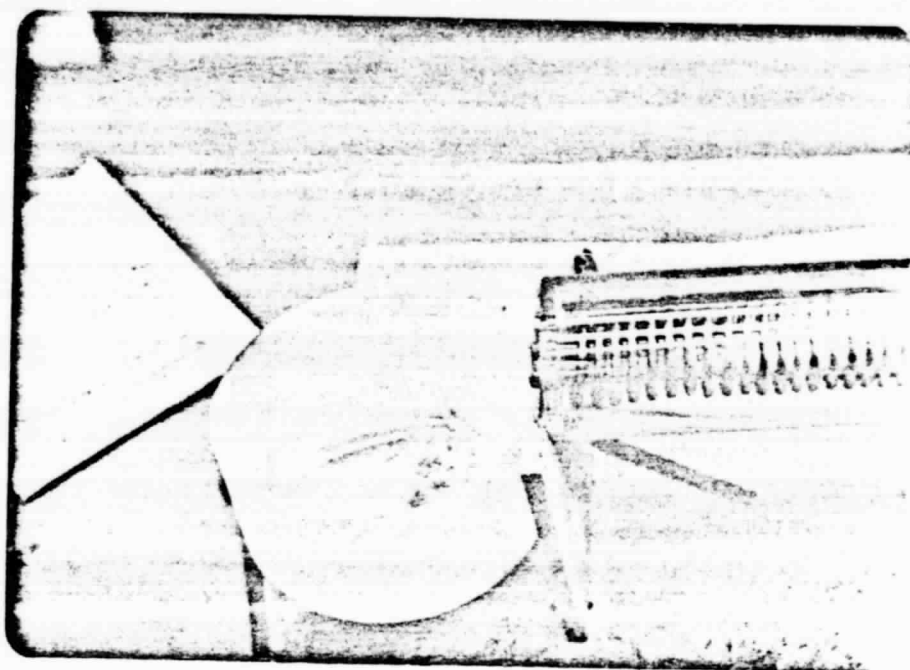


Figure 26



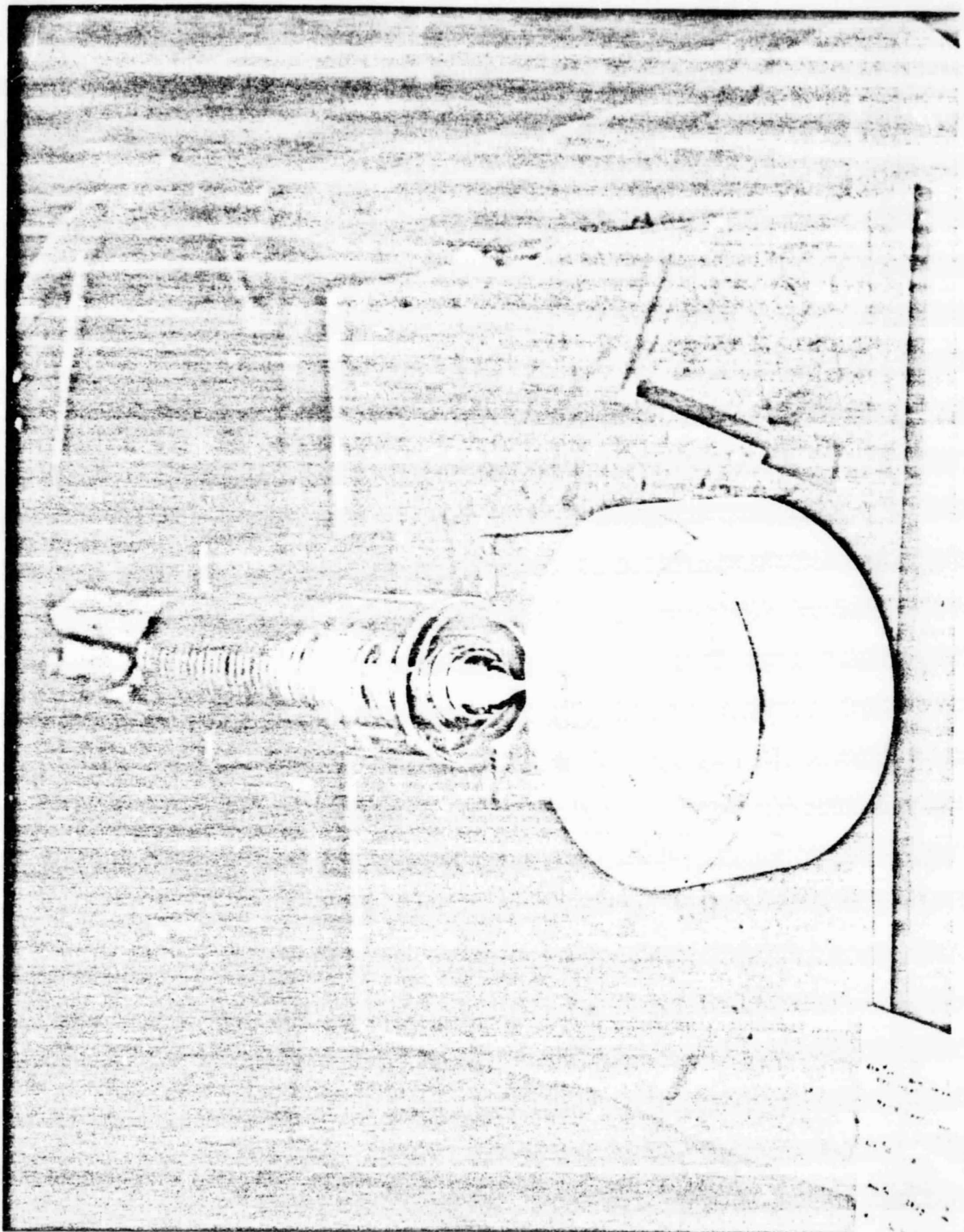
LSA PROJECT
LARGE AREA SILICON SHEET
KAYEX CORPORATION - JPL CONTRACT #955270

Figure 27



Melting in Silver Boat

Figure 28



Melt Exiting From Silver Boat

Figure 29

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reverse side of the silver boat. Supporting the boat just clear of the quartz tube eliminated this problem.

Some problems occurred with solidification at the outlet of the silver boat because of temperature decrease. The RF coil was extended to the outlet of the silver boat to eliminate the problem.

Issuance of the JPL technical direction memorandum relegated the cold crucible to the third priority. The time frame only allowed melt, levitation, and pour experiments to be demonstrated. These experiments were conducted off the puller such that the feasibility of the approach was demonstrated.

The charge material used in the feasibility demonstration was etched recycle silicon size graded to 1/2" to 3/4" OD. This size proved to be satisfactory for adjustment to the RF coil position along the silver boat's length to prevent solidification of the melt at the outlet to the quartz crucible.

A total of eight successful experimental runs were made where melt was transferred into a quartz crucible. Quantities of up to 3 kg were melted with typical power requirements of 3 amp at 9 KV being necessary to achieve melting and transfer.

Further illustrations Figures 28 and 29 show the melt being traversed through the silver boat and melt exiting the silver boat into the crucible.

As previously stated, the feasibility of the approach only was demonstrated by bench-type experimentation. Interfacing of the system to the Czochralski crystal grower could not be done within the contract time frame.

3.0 PROCESS TECHNOLOGY SUMMARY

As previously stated, the issuance of the technical direction memorandum by JPL resulted in new priorities being formulated for the contract as follows:

1. Accelerated meltback of polycrystalline silicon feed rod.
2. Microprocessor control.
3. Accelerated crystal growth.
4. Cold crucible concept for melting lump/granular silicon.

During the course of the contract, two continuous crystal growth runs were performed satisfactorily, namely: 1 x 100 kg at 5" diameter and 1 x 150 kg at 6" diameter.

The initial run was made at the request of the project technical monitor as part of JPL Contract #954888 using the JPL contract #955270 machine. The request was to attempt a 100 kilogram run using a standard unmodified Hamco CG2000 RC puller. The run was successfully completed using a standard 12 inch crucible and ancillary piece parts. A total of 100.3 kilograms was pulled from a total charge weight of 104.5 kilograms. This represents a pulled yield of 96%. A total of 63.9 kilograms of the pulled crystal weight was monocrystalline. This represents a 63.7% yield. The total run time was 108 hours, giving a throughput of 0.93 kg/hr. A series of mechanical problems impacted considerably on the run time and so affected the throughput. Mechanical problems encountered were as follows: (a) crucible rotation stopping due to the drive belt losing several teeth, (b) a broken crystal lift cable due to operator error.

Overall, however, the results obtained confirmed our view that a standard Hamco CG2000 RC puller has the capability of producing 100 kilograms of 5" diameter crystal from one crucible utilizing the hopper silicon recharging device developed on the JPL Contract #954888 program.

The subsequent 150 kg recharge run (Run #30) was made successfully and crystal was grown at a diameter of 6".

A total of 148.5 kg was grown, with a single crystal yield of 45.6%.

The programs as related to the priorities set by JPL are summarized as follows:

1. Accelerated Meltback of Polycrystalline Silicon Feed Rod

The accelerated melt replenishment of the quartz crucible program using an RF coil to melt polycrystalline silicon rod was not successfully developed. The problems encountered were:

- a) Arcing within the growth chamber under a normal vacuum/argon atmosphere operation.

- b) Arcing and electrical breakdown within the RF feedthrough system between the generator and the work coil.
- c) All polycrystalline rods used exhibited cracking problems when heated. Excessive bow and taper in the rods made centering within the coil difficult. Positioning of the rod too close to the RF coil increased the arcing potential.
- d) Purchase of poly silicon feed rods at a cost of \$85 per kilogram made the achievement of the LSA goal of 70¢ per peak watt virtually impossible. No known programs are underway to produce poly rods at a cost of \$14 per kilogram.

It is felt that the technical problems relating to arcing, voltage breakdown, impedance matching, etc. could have been overcome by additional evaluation and development.

2. Microprocessor Control

Microprocessor control of the straight growth process was successfully developed and demonstrated for both 4" and 6" diameter.

Both meltdown and melt stabilization processes were achieved utilizing operator prompting through the microprocessor.

Manual control of the seed, necking processes through the microprocessor were achieved.

Manual taper out of the crystal through the microprocessor after completion of the growth process was achieved.

The effective cost reduction and yield improvement achievable through total automation of the growth process requires additional development.

3. Accelerated Growth Process

The use of the RF work coil used in the poly rod melting program as a heat sink in the accelerated growth program was unsuccessful.

Although acceleration of the growth process was achieved, structure loss problems occurred when the excessive build-up of silicon monoxide on the water-cooled surface of the coil fell into the melt.

The RF coil was replaced in the program by a molybdenum radiation shield. The shield was successful in accelerating the straight growth rate up to an average of 3.3 inches per hour in run number 30 (150 kg continuous recharge run at 6" diameter). Previous average growth rates achieved on continuous recharge run numbers 70 and 72 respectively were 2.84 inches per hour and 2.77 inches per hour. Run numbers 70 and 72 were completed under JPL Contract Number 954888.

Ancillary advantages relating to reduced oxide formation on the crucible wall and elimination of oxide formation on the growing crystal were also achieved.

It is felt that further development of the radiation shield is necessary to achieve optimum accelerated growth rates. This development will achieve the CZ add-on cost reduction projection and improve the monocrystalline growth yield.

4. Cold Crucible Concept for Melting Lump/Granular Silicon

The total design concept for fabrication and interfacing of the total cold crucible system was completed.

The modification to the crystal puller furnace tank to accommodate the cold crucible interface was completed.

The fabrication of the silver boat multi-turn RF work coil and ancillary quartz tubing was completed. Silicon chunk melting, melt levitation and melt transfer experiments were satisfactorily demonstrated by the end of the contract.

There was insufficient time to allow interface of the equipment package into the crystal grower. Further development is therefore necessary to satisfactorily demonstrate the total system.

4.0 ECONOMIC ANALYSIS

SAMICS/IPEG cost calculations have been made during the course of the contract. All the cost calculations have been made using the 1980 dollar figure.

Prior cost calculations and economic models showing the cost per kilogram in terms of CZ add-on cost were made for JPL contract 954888 and are adequately described in section 6 of "JPL Contract Number 954888 - Final Report".⁽¹⁾

For the purpose of this contract report, similar guidelines were used for cost analysis. All SAMICS/IPEG cost data is projected primarily in terms of a "frozen technology" cost analysis. For the purpose of calculating the cost per peak watt, an assumption is made that 1 kilogram of silicon equates to 1 square meter of silicon sheet.

All process development, including continuous recharge growth runs were performed under development and/or prototype conditions. Process runs were made using one Czochralski crystal grower only and the time cycles and supplies costs were extrapolated assuming one operator would run up to 6 crystal growers.

Analysis of the four programs undertaken under this contract reveals that the accelerated growth program was the most effective in terms of economics. Although the contract growth rate goal of 15 cm per hour was not achieved, the actual growth rate achieved did contribute to reducing cost. Ancillary advantages gained from utilization of the radiation shield developed under this program contributed significantly towards improvements in the monocrystalline yield.

As previously stated, further development work is required on both the microprocessor and cold crucible concept programs before the economic and yield projections can be realized.

The SAMICS/IPEG cost calculation shown in Tables 9 through 12 was made to project a CZ add-on cost comparison using polycrystalline silicon rod and polycrystalline silicon chunk material as the start material.

The peak watt cost was calculated also using silicon chunk material start material at a cost of \$65/kg, silicon polycrystalline rod start material at a cost of \$85/kg and silicon lump/granular start material available at a cost of \$14/kg (1986 projected cost). No known cost reduction program is underway to produce low cost polycrystalline rod material; hence, no peak watt cost has been calculated for a process using poly rod as a feed material.

"FROZEN TECHNOLOGY" CZ GROWTH METHODS

CONDITIONS	LOW COST CZ (ROD FEED)	LOW COST CZ (POLY LUMP FEED)
Crucible Size (inches)	14" x 11-1/2"	14" x 11-1/2"
Crystal Diameter (cms)	15.25	15.25
Growth Rate (cm/hr)	15.0	15.0
Total Poly Melted (kg)	160	160
Total Crystal Pulled (kg)	150	150
Pulled Yield (%)	93.75	93.75
Yield After CG (%)	85.0	85.0
No. Crystals/Crucible	5	5
Cycle Time (hrs)	59.8	59.1
Throughput (kg/hr)	2.25	2.28

TABLE 9

SAMICS/IPEG INPUT DATA & COST CALCULATION ASSUMING "FROZEN TECHNOLOGY" STATUS
FOR LOW COST CZ (ROD FEED)/LOW COST CZ (POLY LUMP FEED)

CONDITIONS (PER CYCLE)	LOW COST CZ (ROD FEED)	LOW COST CZ (POLY LUMP FEED)
Total Si Melted (kg)	160	160
Crystal Weight	30	30
No. of Crystals/Crucible	5	5
Diameter of Crystal (cm)	15.25	15.25
Growth Rate (cm/hr)	15.0	15.0
Cycle Time (hrs)	59.8	59.1
Crucible Size	14" x 11-1/2"	14" x 11-1/2"
% Yield (Total in Spec. CG Ground)	85%	85%
Thruput (kg/hr)	2.25	2.25
Input Data (1980 \$)		
Capital Equipment Cost (EQPT)	219,000	209,000
Manufacturing Floor Space (SQFT)	100	100
Annual Direct Labor Salaries		
Prod. Operator (0.65 Persons/Yr)	8,100	8,100
Elect. Tech. (0.3 Persons/Yr)	1,425	1,425
Inspector (0.1 Persons/Yr)	<u>1,068</u>	<u>1,068</u>
Total DLAB	10,593	10,593

TABLE 10

	LOW COST CZ (ROD FEED)	LOW COST CZ (POLY LUMP FEED)
Direct Used Materials & Supplies		
85% Usage Per Year		
Cycles/Yr Hrs/Cycle	124.4/59.8	125.9/59.1
Poly-Kg/Yr (Charged)	19,904	20,144
Seed (\$5.82)	\$ 722	\$ 733
Dopant (Not Costed)		
Argon (100 Ft ³ /Cycle-Hr @ 0.02/Ft ³)	\$ 14,878	\$ 14,881
Crucibles (14" = \$291)	36,084	36,666
Miscellaneous (including graphite:		
\$3.5/Cycle-Hr)	<u>26,037</u>	<u>26,042</u>
Materials Total (MATS)	\$ 77,721	\$ 78,322
Utilities (Process):		
Electricity		
(65kw x 0.035/kw) (Cycle Time - 3 hrs)		
(# Cycles)	\$ 16,075	\$ 16,354
Cooling Water		
(65kw) (\$0.0074) (Cycle Time - 2 hrs)		
(# Cycles)	<u>3,458</u>	<u>3,457</u>
Utilities Total (UTIL)	\$ 19,533	\$ 19,811

TABLE 11

	LOW COST CZ (ROD FEED)	LOW COST CZ (POLY LUMP FEED)
IPEG Price:		
C1 EQPT = \$0.49/Yr - \$EQPT	\$ 107,310	\$ 102,410
C2 SQFT = \$97/Yr - \$SQFT	9,700	9,700
C3 DLAB = \$2.1/Yr - \$DLAB	22,245	22,245
C4 MATS = \$1.3/Yr - \$MATS	101,037	101,818
C5 UTIL = \$1.3/Yr - \$UTIL	<u>19,533</u>	<u>19,811</u>
Annual Cost	\$ 259,825	\$ 255,984
Quan. (Total Charge x % Yield) (kg)	16,918 kg	17,122 kg
Throughput	2.25 kg	2.28 kg
Add-On Cost (\$/kg or \$/m ²)		
(Assume 1 kg = 1 m ²)	\$ 15.36 (1980)	\$ 14.95 (1980)
Projected Costs:		
CZ Add-On Cost Without Silicon	\$ 15.36/kg (0.11¢/peak watt)	\$ 14.95/kg (0.11¢/peak watt)
CZ Add-On Cost With \$85/kg Poly Rod	\$ 138.2/kg (0.99¢/peak watt)	
CZ Add-On Cost With \$65/kg Silicon Lump		\$ 108.8/kg (0.78¢/peak watt)
CZ Add-On Cost With \$14/kg Silicon Lump		\$ 35.2/kg (0.25¢/peak watt)

NOTE: No Projected Cost for Silicon Poly Rod.

TABLE 12

5.0 EFFECTS OF THE PHYSICAL FORM OF SILICON FEED MATERIAL ON THE CRYSTAL GROWTH
PROCESS AND MEASUREMENT OF ANY IMPURITY BUILD-UP IN THE ULTIMATE CRYSTAL PRODUCED

No evaluation or investigation was undertaken during this contract relating to either the effects of impurity build-up in grown silicon crystal or on the effects of the physical form of silicon feed material on the growth process.

General observation suggested that potential thermal cracking of large diameter (> 4 ") polycrystalline feed rods, when suspended over a crucible of molten silicon, created a serious safety hazard. If a portion of rod fell directly into a crucible of molten silicon, it would almost certainly create a "spillage" situation necessitating graphite piece part replacement.

As a result of the "accelerated meltback of polycrystalline rod using an RF work coil" program being de-emphasized, almost all of the work undertaken during this contract was completed using polycrystalline chunk silicon.

Investigation of impurity build-up during multiple growth of silicon ingots was undertaken during JPL contract #954888 Final Report, "Continuous Czochralski Crystal Growth", R. L. Lane and E. G. Roberts ⁽¹⁾ and is considered to be adequately reported. This also applies to impurity build-up within the quartz crucible during the multiple growth of silicon crystals.

6.0 PLANS

During September, 1980, the Kayex Corporation was granted a contract by the Jet Propulsion Laboratory (Contract Number 955733) entitled "Development of an Advanced Czochralski Growth Process to Produce Low Cost 150 kg Silicon Ingots From a Single Crucible for Technology Readiness".

For this contract, it is planned to utilize the hopper recharging techniques developed under JPL contract #954888 for replenishing the growth crucible and also to further develop the radiation shield concept for accelerating the crystal growth process. Further development will also be undertaken to further automate the growth process utilizing microprocessor and sensor techniques.

All of the experience and previously developed technology gained during JPL contract numbers 954888 and 955270 will be incorporated into the design, construction and development of the new CG6000 crystal grower to be used in the JPL contract number 955733 program. The grower will be developed using a 16" diameter crucible capability and be demonstrated with having a technology readiness capability.

REFERENCES

- (1) JPL Contract 954888 Final Report - "Continuous Czochralski Growth", R. L. Lane and E. G. Roberts.
- (2) High Temperature Melting Without Contamination in Cold Crucibles. H. F. Sterling and R. W. Warren. British Journal of Metals, Vol. 67, No. 404.
- (3) Hukin, D. British Patent 1269762 (1972).